SSW-024

Secondary Ion Mass Spectrometric Study of Silver Dissolved in Sulphide Minerals

Volume I of II
Final Report

By

N.S. McIntrye, W.J. Chauvin
and D. Johnston

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Summary

Secondary Ion Mass Spectrometry (SIMS) has been used to quantify trace concentrations of silver and indium within microscopic areas of sulphide minerals. The method was shown to be sensitive enough to detect 1ppm w/w of silver or 0.1ppm w/w of indium. However the accuracy appears to be no better than ±50% at the 100ppm level, because of interferences from molecular ions, surface charging, a contaminated surface region and ion crater edge effects. Most of these effects are correctable and consequently, SIMS analyses are expected to be important in future metal exploration.

Sommaire

On a essayé à faire des analyses, à la fois microscopiques et quantitatives, sur les grains minereaux sulphures, à l'aide d'un Spectrometre de Masse des Ions Secondaire (SIMS). La méthode est sensible à la détection de l'argent au niveau de 1 ppm w/w, et de l'indium au niveau de 0.1ppm w/w. Cependent, l'exactitude de cette méthode, au niveau de 100 ppm ne surpasse plus que ±50%. On croit que ceci est dû aux interférences des ions moléculaires, le chargement electrostatique de la surface, la présence des contaminants dans la couche superficielle de l'échantillon et les contributions vennant du bord du crater fait par le faisceau primaire. La plupart de ces effects peut être corrigée et, par consequent, les analyses fait sur les mineriaux à l'aide de SIMS, doivent ammeliorer à l'avenir.

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Chapter I

Statement of Work

The Cameca IMS-3f ion microscope in Surface Science Western at the University of Western Ontario is to be used to complete the quantitative evaluation of trace element analyses of selected sulphide minerals by SIMS. This particular model of ion microprobe is considered the most technically advanced available with a capability of detecting many elements in the parts per million range. Using standard and mineral samples provided by CANMET, you are to perform the following tasks:

(1) Confirmation of already determined instrumental conditions on standard samples using a beam spot less than 10μm.

(2) Quantitative analysis of up to 750 sulphide mineral grains using high resolution ion microscopy to determine quantitatively Ag and In contents and, qualitatively, other trace elements of interest.

(3) Ion imaging of the sulphide grains to determine if trace elements are homogeneously distributed.

(4) Depth profiles to also ascertain homogeneity.

(5) Complete bar graph mass spectra (to M/e=250).

(6) You will provide access to the laboratory for the Scientific Authority to be present during analyses.

(7) Changes to be made (in details) during the course of the work to reflect new or unexpected results.

Chapter II

Introduction

The recovery of precious metals from mineral ores is frequently hampered by a lack of knowledge of the mineral form within which the precious metal is locked. Thus, the appropriate chemical treatments to effect separation cannot be prescribed. Over 10 million troy ounces of silver were lost in the tailings of Canadian mills in 1980 because of the inability to identify the mineral phase bearing the silver.

Any solution to this problem requires an analytical technique which is, at once, highly sensitive to detect silver in part per million (ppm) concentrations, and at the same time, microscopically discriminating to allow the analysis of mineral grains varying in size from 50-500μm in diameter. In the past, trace element analysis of ores has been restricted to wet chemical, emission spectrographic or neutron activation assays involving crushed composite samples of the entire ore. Microscopic elemental analysis of individual mineral grains has previously depended upon

the use of the electron microprobe, and the detection limit for silver with this technique is ≈0.1% w/w.

Two more recently developed analytical techniques show promise for improved detection of silver within microscopic-size mineral grains. One of these is Particle-Induced X-ray Excitation (PIXE), which uses a focussed high energy (2-4MeV) proton or helium beam to excite characteristic x-rays from most elements in the specimen. The other technique is Secondary Ion Mass Spectrometry (SIMS) which analyzes, with a mass spectrometer all elements removed from the sample surface using a beam of ions, focussed to microscopic dimensions. Both techniques appear to have the potential to determine silver in concentrations as low as 1-10ppm by weight within 200μm diameter regions or smaller. The use of PIXE for such analyses is being explored by the Scientific Authority, while the suitability of SIMS as a trace microscopic technique for mineral analysis has been studied in this Laboratory under contract to the Department of Energy Mines and Resources.

Secondary Ion Mass Spectrometry (SIMS) has, in the past few years, become relatively well-known as a surface analytical technique, capable of detecting elements existing in a wide range of concentrations. This property results from

the high signal/background ratio associated with any mass spectrometric measurement. The microscopic capabilities of the technique are determined, either by the small diameter to which the primary exciting ion beam can be focussed, or alternatively by the use of ion optics to image the source of the secondary ions. The use of SIMS for resolving concentration distributions of trace and minor elements in geologic matrices has been fairly widely recognized, and several good review articles are available (1-3).

It has also been well recognized that quantitative analysis using SIMS is affected by a number of instrumental parameters and sample conditions, many of which are still not fully understood. Secondary ion yields are a strong function of elemental ionization potential or electron affinity, with the result that ion yields vary by several orders of magnitude over the periodic table. Further, the yield is affected by the concentration of oxygen in the matrix and probably by structural differences.

Mass spectral interferences also affect the quantitative reliability of the technique. Molecular ion peaks (frequently oxide or sulphide molecular species of the matrix elements) can contribute to the intensity of a peak being ostensibly measured as a unique elemental species. The relative intensity of molecular ion peaks in the SIMS spectrum is strongly determined by the kinetic energy

distribution of the secondary ions which is, in turn, governed by the extent to which the sample surface accumulates an electrical charge during ion bombardment. Thus particular care must be exercised during the quantitative analysis of non conductors, such as most geologic materials.

Finally, the high surface sensitivity of SIMS can itself cause some difficulties for quantitative analysis. Gradual ion beam penetration of the mineral surface results in some accumulation of redeposited material on the edge of the ion beam crater produced by the beam. Such redeposition on the crater edge can be detected by SIMS if the ion optical system is not exactly aligned.

Despite all the above caveats, quantitative analysis by SIMS should be feasible in a number of situations and is now regularly practiced for some highly demanding analytical problems. Chief among these is the measurement of electrical dopant concentrations in electronic materials (4). In such cases, close matching of standards and specimens is essential. Of particular use is the isotope "spike" technique (5), whereby a known amount of an analyte is introduced into the matrix usually by ion implantation. The SIMS ion current for the implanted element is integrated during an analysis and this is correlated to the known quantity introduced.

This present work statement was proposed following several SIMS experiments at SSW on synthetic silver in pyrrotite ($Fe_{1-x}S_x$) specimens which showed that 1-10ppm concentrations of silver should be detectable by the technique. The work statement proposed that a quantity of 750 different sulphide mineral grains be analyzed for silver and indium during the course of the work which was also to include some time for improvement of the method. The desired analysis points on the grains would be designated by microscopic marks. Several related points on the same grain were usually analyzed to determine if significant variations in silver and indium concentration could be determined across a single grain. An interest in the concentration of indium, as well as silver, stemmed from the hypothesis that the distribution of indium and silver may be related. Substitution of a monovalent silver ion into a ZnS or PbS lattice site may also require substitution of a trivalent ion of comparable size and abundance in the earth's crust into the lattice. Indium is such an element, and, in fact, indium is used as a counter ion in synthetic processes used to produce SIMS silver standards.

The SIMS studies were concentrated on the determination of natural concentrations of silver and indium in two minerals, galena (PbS) and sphalerite ($Zn_{1-x}Fe_xS$) (x=0.1). Ore samples from various Canadian mines were analyzed and

the results compared with synthetic standards of galena and sphalerite which were prepared by the Scientific Authority.

The results of this project are narrated in a chronological order, rather than in any description built around the systematic examination of experimental variables. The reason for a chronological narration is tied to gradual discovery during the project of a number of factors which govern the quantitative SIMS analysis of minerals. The result of this was that only a small number of grains were finally analyzed in a manner felt to be quantitatively satisfactory. In the process, of course, considerable progress was made toward identifying appropriate experimental conditions. A final comparison of PIXE and SIMS analyses of the same mineral grains shows a reasonable collaboration of results, and little indication that, to this point, one technique or the other has a major superiority in accuracy.

Much of the detailed comments on the experiments conducted occurs in the Appendix Section where all scientific correspondence made during the project is exhibited.

Chapter III

Experimental

(a) The Specimens and Standards

SIMS studies were concentrated on the determination of silver and indium in galena and sphalerite minerals. All mineral specimens were mounted in araldite plastic, polished with diamond abrasive and buffed on a cloth lap with a 0.05μm Al_2O_3 slurry. Frequently standards and unknowns were placed in the same block. The standards were prepared by solid state high temperature reactions of the ingredients in a sealed tube. Both standards and unknowns were prepared by the Scientific Authority at the Canada Centre for Mineral and Energy Technology. The methods of preparation are described in detail elsewhere (6).

(b) SIMS Analysis

A Cameca IMS-3f ion microscope was used for all SIMS analysis. This instrument had a mass-filtered primary ion beam which was either 8 kV O_2^+ or 8 kV O^-

ions. O^- primary ions were used through most of the study since better charge neutralization was achieved than with the O_2^+ beam. The beam current employed is roughly inversely proportional to beam diameter. Beam currents of <10nA are required to produce diameters of 10-20μm, while beam currents of 400-800nA can be used for diameters of 40-50μm. Initially, non-rastered primary beams were used in order to achieve high spatial resolution, but in later work the beam was rastered over a 250x250μm range. The field area accepted by the secondary ion spectrometer was some fraction of the beam diameter, usually 60% or less. Thus, for a 15μm beam, an area of ≈10μm was selected.

The kinetic energy distribution of secondary ions measured was determined by the "offset" voltage placed on the sample stage. Normally, no offset voltage was applied and the kinetic energy range (window) of ions accepted into the analyzer was 150eV. In some experiments the relative molecular ion component in the spectrum was reduced by using an offset voltage of 75 volts and a window of 50eV. The use of such "offset" voltages in geochemical SIMS analysis has been reviewed by Shimizu and Hart (7).

Several different procedures for ion counting were used. Initially, the computer routine "Isotope" was used to collect and time-average the ion intensity ratios. In later stages of the program a running average of the ratio was monitered because of the need to detect changes in concentration during the course of the analysis in one location.

To reduce the surface charging effects, a thin (100nm) layer of gold was deposited on the surface before SIMS analysis was carried out.

Chapter III

Results

(a) Preliminary Studies (July 1982-September 1983)

In this period (not covered by contract) the feasibility of detecting silver at trace levels was confirmed (see Appendix - A-1) and initial quantitative measurements were made on some trial PbS synthetic "reference" materials (A-2, A-3, A-4) supplied by the Scientific Authority. SIMS studies of these silver-doped lead sulphides were carried out with a non-rastered primary O_2^+ beam of 60µm diameter. The intensity of the $^{107}Ag^+$ ion was ratioed to the $^{208}Pb^+$ peak and a reasonably linear correlation was found for five specimens containing 0.001-0.05% w/w Ag (see Figure 2 in A-3). These first runs showed that a silver detection limit of ≈2 ppm could be expected with the O_2^+ beam conditions used on a galena matrix.

Subsequent work with sphalerite reference materials (A-2) mounted in araldite indicated that specimen charging was a more serious problem than with

galena. One solution to this was to mount the minerals in a conducting metal matrix (Cerrotu alloy). Unfortunately, polishing of this type of mount transferred trace amounts of silver and cadmium from the Cerrotu alloy into the mineral surface. Thus, use of araldite as a mounting material was maintained and experimentation with small O^- primary beams began.

The use of an O^- beam (50μm diameter) was found to result in improved control of surface charging and therefore a series of doped synthetic sphalerites were analyzed in July 1983, and reported in September 1983 (A-5). Four doped "reference" materials, with nominal silver concentrations ranging from 0.011% w/w to 0.11% w/w, were found to give $^{107}Ag^+/^{66}Zn^+$ ion intensity ratios which varied with these nominal compositions in a reasonably linear relationship (see Figure 2 of A-5). A blank synthetic sphalerite was also analyzed with the group and, by linear extrapolation of the remaining data points, a silver concentration of ≈20ppm was predicted.

Thus, at this point in the study, it appeared that, at the very least, a rough correlation could be drawn between the SIMS ion intensity ratio and the concentration of trace silver in sphalerite or galena.

(b) Effects of Primary Beam Parameters (September 1983-March 1984)

The subsequent stages of the project involved investigations of (among others) the effects of beam intensity, beam size, and specimen preparation techniques. In addition, the first analyses of natural sphalerites were undertaken. Two araldite blocks were received (B-1), each one containing a series of pieces of the previously-synthesized galena (labelled Block A) and sphalerite (labelled B) materials. In addition, Block B contained several natural sphalerites from the Nanisivik, N.W.T. mine were mounted.

The synthetic sphalerites were analyzed with a large (>300μm) non-rastered O^- beam and a field aperture area of 150μm. Both In^+/Zn^+ and Ag^+/Zn^+ ratios were correlated to nominal bulk compositions. The linear correlation for both doped elements was excellent - a significant improvement over previous attempts (see Figure 1 in B-2).

The effect of measuring a much smaller area on the specimen was investigated by decreasing the field aperture of the SIMS instrument to measure an 8μm area, instead of 150μm. The result of this area reduction was a substantial decrease in both In^+/Zn^+ and Ag^+/Zn^+ absolute ratios - a factor of three, but no change in the slope of the analytical curve. Such a major change in intensity could have resulted from the necessity of opening the instrument contrast aperture to gain enough intensity for measurement of the low silver ion intensity. This change in contrast aperture affects the kinetic energy distribution sensed by the mass analyzer. Thus, the larger contrast aperture may result in more molecular ion suppression around masses 107 (Ag) and 115 (In) with proportionally lower Ag/Zn and In/Zn ratios.

The reproducibility of the above ratios was examined over a period of several days. Absolute ion values were found to change by ±10% from day to day, but similar changes were found for all points. Thus the reproducibility of the slope was somewhat better than that of the individual point values. The variation in ion ratio is believed to be related to the magnitude of the molecular ion contribution at mass 115

or 107. This, in turn is governed by changes in the kinetic energy distribution, and this could be affected by charging of the surface. Thus, it was necessary to rerun a calibration graph each day after the instrument has first been adjusted. Indium was detected with a sensitivity of ≈50 times greater than that of silver. Therefore, assuming similar backgrounds at masses 115 and 107, the detection limit for indium in sphalerite was determined to be ≈0.1 ppm w/w.

The effect of surface finish on the impurities left behind was investigated briefly. No changes were noted in the apparent blank silver or indium concentrations with changes in polishing techniques, even though the quantity of other elements present was affected markedly by polishing. The amount of silver background in "blank" synthetic specimens continued to be a problem. Blanks prepared by different methods gave differing silver content when analyzed by SIMS.

Analysis of the Nanisivik sphalerite grains revealed strong changes in silver content from sample to sample, and even, in some cases within a grain itself (B-2). Such silver concentration distributions were however, still too weak to be detected by ion imaging.

The experience gained to this point was tested by analyzing a block (labelled WA) containing 38 natural sphalerite specimens (see B-4). Each specimen had several 100μm diameter areas designated by the Scientific Authority, and within each such areas, several analyses were made. The areas are illustrated in the photograph in B-5. In order to achieve this spatial resolution, the primary O^- beam size was reduced to a size 10-20μm in diameter (1nA primary beam current). The other means of achieving a small area analysis, use of a small field aperture (see above) could not be employed since a number of separate analyses were required within a very close distance of each other.

The hundred odd analyses done in this series extended over the period of a number of days, and each day a new calibration graph was obtained. Therefore, the large amount of data on the synthetic materials provided better evidence of the extent of their validity. The data scatter for a series of different calibration runs is seen in Figure 3 of B-5, and the magnitude of day to day variation for both In and Ag confirms the earlier data (≈±10%). A study of the reproducibility arising from two discrete areas of each synthetic specimen (Figure 2 of B-5) shows variation

similar to that in Figure 3, except that the lowest two concentrations of silver-doped materials appear rather inhomogeneous.

The analyses of the unknown sphalerites gave silver concentrations ranging from 10,000-30 ppm and indium concentrations from 2000-8ppm. In general, there was little correlation between the locations of elevated silver and indium concentrations.

During SIMS analysis of the natural sphalerite grains, the Ag^+/Zn^+ and In^+/Zn^+ ratios did not completely stabilize with time as the ion beam penetrated the mineral. Rather, the ratios measured continued to decrease slowly, to the extent that, after the first ten minute measurement period, ratios decreased by as much as a factor of two during the next five minute measurement period. This slow approach to an "equilibrium" value is now ascribed to an altered surface layer and to sputter redeposition around the periphery of the crater.

(c) Investigations of Precision and Accuracy in the SIMS Analysis of Natural Sphalerites (April 1984 - May 1985)

Various theories were advanced to explain the decreasing response of silver and indium in the natural sphalerites included as part of the WA block. Surface enrichment of these two species due to polishing or ion beam heating were suggested (C-2). Later (C-3) following comparison with data obtained on the Block B sphalerites, sputter redeposition (see above) was recognized as another possible contribution. This was confirmed (C-4) by SIMS analyses repeated on Block WA, this time using a 40μm O^- beam. Less of a decrease in relative silver and indium ion intensities was found. The average results of this run agreed only within a factor of two with previous analyses on the same specimen. Analyses of Block B natural sphalerites (C-6) also confirmed this. It was also shown that a typical 150 second analysis with a 60nA (40μm diameter) O^- beam penetrates only 100nm (0.1μm) into the surface. The smaller (10-20μm) beam used in the first analyses of the WA series would have penetrated less than 20nm into the surface.

The sphalerite Block WA was repolished by the Scientific Authority (C-5) and renamed Block WB-sphalerite. A number of these same natural sphalerites were reanalyzed using a 40μm O^- beam (C-8). SIMS depth profiles were run on a number of mineral surfaces to show that, although silver, indium, and cadmium intensities are particularly enhanced in the outer 50nm of the polished surface, all values reach a steady state at beam penetration depths greater than this (see Figure 3 of C-8). Thus, the beam must be intense enough to penetrate the anomalous surface "crust" or skin formed during polishing and, as well, be large enough to avoid redeposition effects from being detected by the wide open secondary column of the SIMS. The new silver and indium concentrations were tabulated for the natural sphalerites in Table 2 of C-8.

The consequence of determining very low silver concentrations was explored using natural sphalerite specimens from Block A(25/10/83). Concentrations of less than 1ppm w/w of silver were detectable, but the uncertainty involved in the measurement is enormous because of the lack of any reliable reference value below 10ppm in concentration.

Following the repolishing of Block W-A(C-10), both it and Block B sphalerite were again analyzed for the synthetic standards (C-11). Analytical curves were obtained for Ag^+/Zn^+ and In^+/Zn^+ with and without a kinetic energy offset voltage of 75eV applied. Although the ion intensity ratios decrease by a factor of ≈10 with offset voltage applied, the slope of the calibration curve and the scatter of the data points (correlation coefficient) were unaffected.

Nonetheless it was felt that the use of kinetic energy offset may be of particular use in analyzing natural sphalerites where molecular ions due to unknown components may affect the ion intensity at masses 107 or 109. Consequently a series of natural sphalerites was analyzed with and without offset voltage applied. Further, the effect of changing instrumental operators was also investigated. The silver and indium values for these two alterations were recorded on Tables 2 and 3 of C-15 and are compared with PIXE and electron microprobe analyses of some of the same grains. Non-offset conditions gave results which were in slightly poorer agreement with PIXE values. Also, the techniques of a particular operator appear to have some effect on the results. Finally, inhomogeneity within the specimen does lead to some serious discrepancy.

SIMS depth profiles of two specimens (3 and 4) showed these to have highly variable silver concentrations, and indeed SIMS analyses for these two specimens, showed poor correlation with PIXE data. In the other 10 cases for silver data where comparisons are possible 7 of the comparisons agreed within the precision of the measurements.

A series of Ag and In calibration curves for synthetic sphalerites referenced to iron were also obtained, and the correlation coefficients were better than for curves referenced to zinc. This may yield better results in future if an accurate value for the iron content can be obtained independently.

(d) Precision and Accuracy in SIMS Analysis of Natural Galenas (September 1983-February 1984)

Synthetic and natural galena specimens in Block W-C (D-1) were analyzed for silver concentration. Reasonable silver homogeneity was found in the synthetic "reference" materials. In the natural galenas, many of the points analyzed did not vary sharply in silver concentration with depth. However some points with particularly low silver concentrations did exhibit the same rather monotonic decrease described in C-5. We

attribute this to contamination of the top skin of the polished layer with silver form other specimens. It is important that this skin be cleanly penetrated by the beam in order to effect an accurate analysis of the underlying mineral with a low silver content. Apart from this, the data are quite interesting and probably afford the most reliable and intensive survey of local variations in silver concentration. This variation can sometimes be observed on a submicron scale through the information given during a consecutive series of SIMS analysis (see 2D in D-1, for example). Also, point analyses within the circled areas show significant variation in silver content.

Chapter IV

Discussion and Conclusions

Discussion

Through a complex and often confusing series of analyses, several factors affecting accuracy and precision have been identified.

The contribution of molecular ion intensity to that of the ion representing the trace analyte is large enough to change significantly to the ion signal at low (<100ppm) silver concentrations. More important, the contribution of molecular ion intensity is altered by relatively minor changes in ion kinetic energy distribution. This distribution is influenced by the extent of charging on the mineral surface. Even minor (<20eV) changes in charge distribution on this insulating surface can therefore alter the contribution of molecular ions entering the analyzer. Surface charging of this magnitude is affected by factors such as primary beam density, and the electrical conductivity of the specimen. This latter factor can itself be affected by the nature of the electrical path between the surrounding gold coating and the crater bottom. In addition, differences in the conducting nature of the polished outer skin of the mineral and the underlying mineral phase can cause a change

in the surface charging. The nature of the crystal structure of the mineral will also affect charging and it may well be that the degree of substitution of impurities will also play an important role.

In general, the charging effects are subtle and their influence remains at a level which usually allows reasonable quantitative measurements at the 100-200ppm level (i.e. ±10% at 200ppm). This degree of reproducibility is observed in both series of synthetic minerals analyzed.

It is clear that better reproducibility will result from an approach that takes care to maintain all conditions of the analysis very constant (beam current, area chosen for analysis etc.). It is also clear that reliance on doped synthetic materials as standards may be misleading, since the conducting properties of these materials could differ from those of the natural minerals. The current attempts by S. Chryssoulis (8) to develop the internal spike method for standardization should provide a more reliable means of determining precious metal elements in mineral matrices of varying electrical and compositional properties. However the degree of doping by ion implantation must be kept at a level low enough not to alter significantly the conducting properties of the surface.

Can charging effects be counteracted in other ways? One possibility is to minimize the molecular ion contribution in the fist place by working at high offset voltages (+125eV in the Cameca IMS-3f) and with reduced energy slits. Since silver ion intensities are not usually the limiting factor in the analysis, this seems like a reasonable prospect. The other more uncertain possibility is that the kinetic energy distribution of a major ion be monitored frequently during the analysis. Since the slope of the secondary ion kinetic energy curve is very large on the low energy side of the distribution (9) quite accurate (±5eV) measurements of charging can be made. Such measurements are, however, time-consuming.

A second factor affecting quantitation is the presence of surface polishing artifacts. The outer skin of the mineral contains analyte contamination transferred during polishing from neighboring grains. The conduction properties (see above) also differ from the underlying mineral. In general, the skin appears to be as much as 50nm deep, and primary beams must therefore have sufficient current density to penetrate the skin completely in a reasonable time.

Finally, it has become clear that quantitative analysis must be carried out on an area well removed from the ion

beam crater edge. Thus the use of small area beams (<50μm diameter) and even non-rastered beams are likely to continue to add to the uncertainties involved.

To conclude on a positive note, it appears that SIMS has quantitative capabilities close to those of PIXE at the 100ppm level for silver in sphalerite. Further, the detection limits of both silver and indium appear to be somewhat better with SIMS than for PIXE. Finally the surface sensitivity of SIMS gives it a submicron microanalytical capability as the beam passes through concentration gradients within the mineral. This is best illustrated by the profiling of silver in galena as shown in D-2.

Conculsions

(1) SIMS has been shown capable of detecting trace levels of silver and indium in the sulphide minerals sphalerite and galena. The detection limits for silver and indium are 1ppm w/w and 0.1ppm w/w respectively.

(2) Using external reference materials, a number of natural minerals have been analyzed with modest accuracy (±50% at 100ppm) using primary oxygen ion beams of 50μm diameter.

(3) The method is susceptible to interference from molecular ion contributions. Such contributions can be minimized by limiting analysis to ions with kinetic energies >100eV.

(4) Surface charging is another source of inaccuracy and control of this be improved.

(5) Crater edge effects limit the size of area analyzed to that of 50μm or greater. While this places limitations on the study of the local variations of a trace element, some measure of the variation can be obtained from the change in ion intensity as a function of time as the primary beam penetrates the mineral.

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SSW-024-Appendices

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Appendices

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Appendices

A - Preliminary Studies (July 1982 - September 1983)

B - Effects of Primary Beam Parameters (September 1983 - March 1984)

C - Investigations of Precision and Accuracy in the SIMS Analysis of Natural Sphalerites (April 1984 - May 1985)

D - Precision and Accuracy of SIMS Analysis of Natural Galenas (September 1983 - February 1984)

A

PRELIMINARY STUDIES

(July 1982 - September 1983)

A-2

3C.

The University of Western Ontario

Centre for
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in Chemical Physics
Faculty of Science
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July 8, 1982

Dr. L. Cabri
Canada Department of Energy
Mines and Resources
568 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Dr. Cabri:

I am enclosing Secondary Ion Mass Spectra taken of a number of your standards and sulphide specimens transmitted to us by Dr. Rod Packwood. These spectra were taken by Mr. Bill Chauvin of our group and were analyzed by he and I. The ion microprobe was run using a 200 um wide beam of 15KeV positive oxygen ions.

Our main objective was to survey as many of the samples as possible, so as to determine which elements showed good promise for trace determination in the sulphide matrix. As such, refinements such as high mass resolution and kinetic energy analysis were not used for the present. Specimens 1,3,4,5, 6,8,9,10,11,13 and 15 were analyzed and these are discussed below, with those containing a particular element grouped together.

Group 1 - Palladium - Spectrum 1-1 is a mass scan of the palladium isotopes. It can be seen from the log intensity scale that the intensity of each major isotope is several orders of magnitude above background. Figure 1-2 is a "bar graph" mass spectrum of sample 3 (FeNiPdS). The iron and nickel form ionic clusters of oxygen and sulphur and one of these ($FeSO^+$) falls directly over the palladium isotopes making it impossible to identify them in this specimen under these instrumental conditions. We probably could separate the Pd and $FeSO^+$ peaks under higher mass resolution (mass defect differences) without a substantial drop in intensity. My guess however is that the practical detection sensitivity would not be better than 1000 ppm (w/w) in this matrix, even under high resolution.

Conclusion - Not promising for trace determination, at least with an O_2^+ primary ion beam. A cesium beam may be better for this particular matrix.

Group 2 - Silver - In lead sulphides, silver can be well detected at the trace level, as can be seen from Figures 2-1 and 2-2. I would estimate a detection limit of 1-10 ppm under these conditions. Thus, employing longer counting times and somewhat higher resolution, a detection of 0.5 ppm should be quite possible. Even FeS would be an acceptable matrix.

A-1 (cont)

Conclusion - a very good prospect.

Group 3 - Rhodium in Iron Sulphide. Figure 3-1 for synthetic rhodium pyrrhotite shows that the ^{103}Rh intensity is very strong. Even in a predominently FeS matrix, mass 103 is clear (see Figure 1-1 [C]) and in the specimen containing 5000 ppm ^{103}Rh is clearly seen. Based on the mass 103 background measured in Figure 1-1 a detection limit of 10 ppm rhodium appears possible under these conditions. Thus by squeezing down the resolution or the kinetic energy filter we should obtain an ultimate limit of 2-5 ppm in this matrix. Incidentally, $^{206}Pb^{++}$ would interfere, probably severely, so a lead isotope would have to be monitored constantly.

Group 4 - Iridium - Figure 4-1 shows the isotope distribution for a platinum-iridium alloy. The iron sulphide specimen containing 1200 ppm iridium is shown in Figure 4-2. Fortunately, ^{191}Ir is not interferred with by an $FeSO^+$ cluster (in this case, $Fe_2S_2O^+$) and iridium is possibly detected in this spectrum. In this situation, it may be possible using kinetic energy analysis to suppress the cluster ions without affecting the iridium yield. Under such conditions one may reach a detection limit of 100-500 ppm Ir in FeS (or significantly lower in a galena). Again, a cesium beam may be better for production of Ir^+ and the production of $FeSO^+$ cluster ions would be reduced significantly.

Conclusion - not too sensitive under the conditions tried.

Group 5 - Selenium - Figure 5-1 shows a spectrum of elemental selenium. Figure 5-2 shows the spectrum of 1700 ppm Se in FeS. The selenium cannot be identified above the background.

Conclusion - poor detection limit under these conditions. Cesium primary ions would produce much better results - increasing the yield and drastically reducing the $FeSO^+$ clusters.

In summary, it appears that under the present O_2^+ primary ion condition, detection of silver and rhodium is very good in these matrices, palladium and iridium are definitely lower in sensitivity, while selium is very low indeed.

I look forward to your comments.

Yours very truly,

N.S. McIntyre
Technical Director
Surface Science Western

cc: Mr. Bill Chauvin

A-2.

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

Research and Technology
Recherche et technologie

Canada Centre for Mineral and Energy Technology,
555 Booth Street
Ottawa, Ontario
K1A 0G1

Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa (Ontario)
K1A 0G1

Your file Votre référence

Our file Notre référence

REGISTERED AIRMAIL

January 20, 1983

Dr. N.S. McIntyre,
Technical Director,
Surface Science Western,
Centre for Interdisciplinary Studies
in Chemical Physics,
University of Western Ontario,
London, Ontario
N6A 3K7

Dear Stewart:

I am happy to enclose our first set of PbS standards doped with Ag for our joint project as discussed on the phone and by letter. I believe that all samples have sufficient large areas free of pits and imperfections, except maybe for one. I will be very interested in your results and, as I will be out of town Feb. 7 to March 1, would be grateful if you have something to report before Feb. 7. Details of the standards are on the enclosed sheet.

We are proceeding well with the ZnS standards, after a lot of experimental failures. These are doped with Fe, Ag and In. They are presently "cooking" at 900°C.

With best wishes.

Yours sincerely,

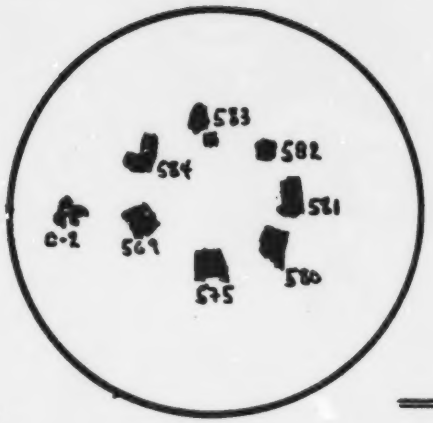
Louis J. Cabri

Encl.
cc:J.H.G. Laflamme

LJC/tj

Canada

A-2 (cont'd)



Wt% (weighted-in values)

	Pb	Ag	S
C-2	86.20	0.40	13.40
569	86.60	0.00	13.40
575	86.50	0.10	13.40
580	86.55	0.05	13.40
581	86.575	0.025	13.40
582	86.59	0.01	13.40
583	86.595	0.005	13.40
584	86.599	0.001	13.40

A-3 Canmet.

The University of Western Ontario

Centre for
Interdisciplinary Studies
in Chemical Physics
Faculty of Science
London, Canada
N6A 3K7

Telephone (519) 679-6371
Telex UWO LIB LDN 064 7134

February 15, 1983

Dr. Louis J. Cabri
Canada Centre for Mineral and
Energy Technology
555 Booth Street
Ottawa, Ontario
K1A OG1

Dear Louis,

I enclose the results from our ion microscopic studies of the set of PbS standards which you sent us.

Specimens were analyzed with a 12 KeV beam of O_2^+ with a diameter of about 60 um. Preliminary studies showed that counting times per isotope of 10 seconds or greater would be required for adequate statistics. Also, an anomalous composition near the surface of each specimen had to be penetrated before reproducible data was obtained. Figure 1 shows a depth profile of $^{107}Ag^+$, $^{32}S^+$, $^{27}Al^+$, and $^{208}Pb^+$ through the surface region. Could this surface alteration be due to impurities from the polishing wheel or from surface heating during polishing?

Subsequent studies of each specimen were done after the surface had been sputtered for several minutes to remove any anomolies. Ten sequential twenty second counts were taken on both $^{107}Ag^+$ and $^{208}Pb^+$ peaks and the silver intensity was ratioed to that of the lead.

The intensity ratios are given below for each specimen, along with the 2σ precision calculated for a single twenty second count. Rough supporting data is also included.

Specimen	Concentration	Ag/Pb Ratio
C-2	0.4% Ag	$6.4 \pm 1.6\ (2\sigma) \times 10^{-3}\{\sigma = 12\%\}$
569*	0% Ag	$1.1 \pm 0.46(2\sigma) \times 10^{-5}\{\sigma = 21\%\}$
575	0.1% Ag	$1.0 \pm 0.4\ (2\sigma) \times 10^{-3}\{\sigma = 20\%\}$
580	0.05% Ag	$4.1 \pm 1.4\ (2\sigma) \times 10^{-4}\{\sigma = 15\%\}$
580 $\{I_2\}$*	0.05% Ag	$4.5 \pm 0.3\ (2\sigma) \times 10^{-4}\{\sigma = 4\%\}$
581	0.025% Ag	$2.75 \pm 0.3\ (2\sigma) \times 10^{-4}\{\sigma = 6\%\}$
582	0.01% Ag	$1.3 \pm 0.3\ (2\sigma) \times 10^{-4}\{\sigma = 13\%\}$
583	0.005% Ag	$1.1 \pm 0.3\ (2\sigma) \times 10^{-4}\{\sigma = 18\%\}$
584	0.001% Ag	$3.5 \pm 0.4\ (2\sigma) \times 10^{-5}\{\sigma = 10\%\}$

* Used for background correction; $\{I_2\}$ - larger beam diameter used

H-3 cont'd.

The beam current used in these experiments was around 800 nA. Experiments showed that much higher beam currents (~ 4500 nA) did not noticeably improve the relative sensitivity of the measurement or its precision. Moreover, change in the beam size used had no effect on the sensitivity.

If the intensity ratio for specimen 569 is assumed to be due to background only, then it can be subtracted from other results. The result of this are shown in the graphs on Figures 2 and 3. Aside from specimen C-2, a linear correlation between intensity ratio and concentration is quite apparent. A detection limit of 2 ppm is calculated, based on three standard deviations above the mean value of the noise in the background signal from specimen 569. If, however, the background specimen (569) contains trace quantities of silver, the detection limit would be lower.

We therefore conclude that there is a good possibility of using ion microscopy for quantitative detection of trace level silver in sulphide ores. Newer approaches with SIMS which we are presently testing, may allow us to reduce the detection by an order of magnitude.

I note from your letter that you are preparing other specimens to submit to our Laboratory. My understanding was that we would undertake only this present series at no cost. Any subsequent studies will be charged at our normal commercial rates.

I hope that these results meet your expectation and that we can be of service to you in future.

Yours very truly,

N.S. McIntyre
Project Manager
Surface Science Western

cc: J.H.G. Laflamme
W.J. Chauvin, UWO

G-14 SQUARE 10 X 10 TO THE CM
SPECIFY TRACING OR DRAWING PAPER
GRAPHIC CONTROLS CANADA LTD.
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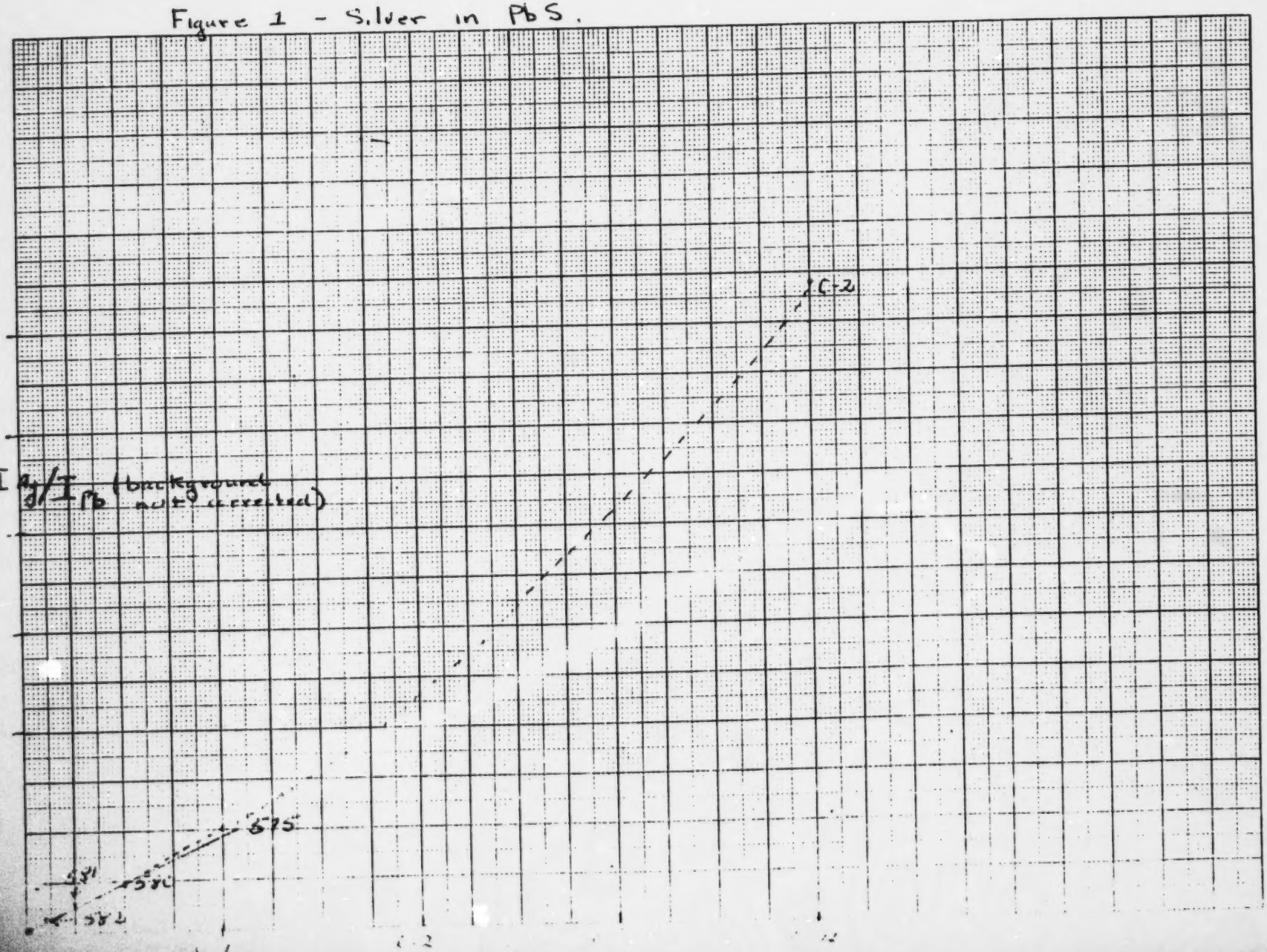
Figure 1 - Silver in PbS.



4-3 (cont'd)

G-14 SQUARE 10 X 10 TO THE CM
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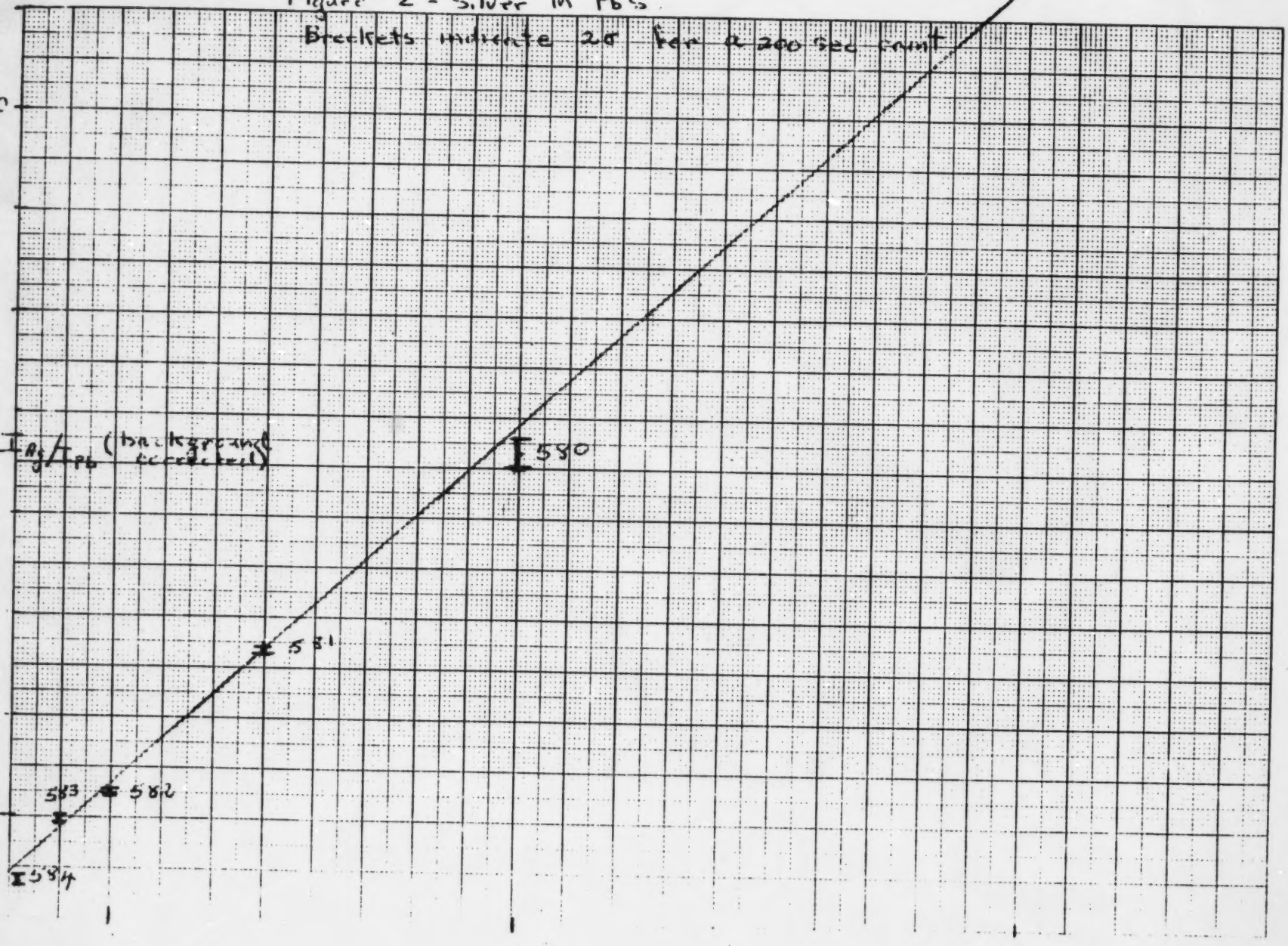
Figure 2 - Silver in PbS

Brackets indicate 2σ for a 200 sec count



A-3 (cont'd)

Ag in PbS H-3 (cont'd)

Second Series

Conditions: Ion Current = 7½ mA

Count times = 20 sec.

Specimen Cal - 0.110 % Ag

$^{111}Ag/^{206}Pb$

7.6×10^{-3}

6.0×10^{-3}

5.4×10^{-3}

5.3×10^{-3}

5.6×10^{-3}

$\sigma = 0.5\ (12\%)$ For Std (10 [illegible]

5.9×10^{-3}

6.3×10^{-3}

6.1×10^{-3}

6.4×10^{-3}

7.1×10^{-3}

Av: 6.41×10^{-3} R: [illegible]

STD = [illegible]

Specimen Cal - 0.0% Ag

$^{111}Ag/^{111}Pb$

$\times 10^{-5}$

1.1×10^{-5}

1.2×10^{-5}

1.1×10^{-5}

1.2×10^{-5}

$\sigma = 0.23\ (21\%)$

$3\sigma = 0.7$

[illegible] Std $\sigma = 0.02$

0.1×10^{-6}

1.3×10^{-5}

0.1×10^{-5}

1.0×10^{-5}

1.3×10^{-5}

Av: 1.1×10^{-5} $\sigma = 0.25\ (21\%)$

STD = [illegible]

$3\sigma = 0.7$

H-3 (cont'd)

Specimen .75" - 0.10 %

Ag 11b 8.2 × 10⁻⁴
Blk #1 8.7 × 10⁻⁴
9.0 × 10⁻⁴
7.7 × 10⁻⁴
8 × 10⁻⁴

Av = 8.5 × 10⁻⁴

σ = 0.2 (20%)

Blanks run separately

Blk #1 1.5 × 10⁻³
1.3 × 10⁻³
1.1 × 10⁻³
1.0 × 10⁻³
1.0 × 10⁻³

Av = 1.2 × 10⁻³

Grand Av = 1.0 × 10⁻³

Specimen 38" - 0.05 %

Ag 11b 3 × 10⁻⁴
2.0 × 10⁻⁴
4.1 × 10⁻⁴
4.0 × 10⁻⁴
4.1 × 10⁻⁴

For 7 [illegible]
σ = 0.7 (15%)
For 5 [illegible]
σ = 0.01

4.1 × 10⁻⁴
5.0 × 10⁻⁴

Av = 4.7 × 10⁻⁴
Range 2:1

← results
← rejected.
Blanks out of specimen.)

H-3 cont.

(3

Specimen ... 581 (I2) - 0.05% C

... with the discussed ...

7.4 × 10⁻⁴ → Reject
6.7 × 10⁻⁴ → Reject
5.3 × 10⁻⁴
5.5 × 10⁻⁴
4.3 × 10⁻⁴

For ... results
σ = 0.17 (6%)
For standard ...
σ = ...

4.4 × 10⁻⁴
4.3 × 10⁻⁴
4.2 × 10⁻⁴
3.9 × 10⁻⁴
4.0 × 10⁻⁴ Av = 4.15 × 10⁻⁴

3 9

Specimen 581 (I_1) - 0.025% C

2.4 × 10⁻⁴
2.6 × 10⁻⁴
2.7 × 10⁻⁴
2.8 × 10⁻⁴
3.0 × 10⁻⁴

σ = 0.17 (6%)
For standard (10 ...
σ = 0.02

2.5 × 10⁻⁴
2.8 × 10⁻⁴
2.8 × 10⁻⁴
2.6 × 10⁻⁴
2.9 × 10⁻⁴ Av = 2.75 × 10⁻⁴

Range = 0.6

H-3 (cont'd)

Specimen 532 - 0.01%

1.1 × 10⁻⁴
1.1 × 10⁻⁴
1.3 × 10⁻⁴
1.2 × 10⁻⁴
1.3 × 10⁻⁴

σ = 0.17 (17%)
For standard (1%)
σ = .02

1.3 × 10⁻⁴
1.3 × 10⁻⁴
1.4 × 10⁻⁴
1.5 × 10⁻⁴
1.4 × 10⁻⁴ Av. rate 1.3 × 10⁻⁴ Range = 4

Specimen 533 - 0.005%

8.4 × 10⁻⁵
9.9 × 10⁻⁵
8.1 × 10⁻⁵
1.1 × 10⁻⁴
1.4 × 10⁻⁴

σ = 0.18 (18%)
For standard (1%)
σ = 0.02

1.1 × 10⁻⁴
1.2 × 10⁻⁴
1.3 × 10⁻⁴
1.1 × 10⁻⁴
1.1 × 10⁻⁴ Av. = 1.1 × 10⁻⁴

Specimen 584 -0.001%

2.6 × 10⁻⁵ ⇒ Reject
2.7 × 10⁻⁵ ⇒ Reject.
3.0 × 10⁻⁵
3.2 × 10⁻⁵
3.3 × 10⁻⁵

σ = 0.35 (1σ)
For standard / sample.
σ = 0

4.1 × 10⁻⁵
3.4 × 10⁻⁵
3.7 × 10⁻⁵
3.7 × 10⁻⁵
3.9 × 10⁻⁵

∴ 3.5 × 10⁻⁵ Range =

Summary

Specimen	Concentration	Ag/Pb Ratio
C-2	0.4% Ag	6.4 ± 1.6 (2σ) × 10⁻⁵
564 *	0% Ag	1.1 ± 0.46 (2σ) × 10⁻⁵
575	0.1% Ag	1.0 ± 0.4 (2σ) × 10⁻⁵
580	0.05% Ag	.47 ± 1.2 (2σ) × 10⁻⁵
580 [I₂]	0.05% Ag	4.5 ± 0.3 (2σ) × 10⁻⁵
581	0.01% Ag	2.75 ± 0.3 (2σ) × 10⁻⁵
582	0.005% Ag	1.3 ± 0.3 (2σ) × 10⁻⁵
583	0.003% Ag	1.1 ± 0.3 (2σ) × 10⁻⁵
584	0.001% Ag	3.5 ± 0.4 (2σ) × 10⁻⁵

* used for background correction; [I₂] - larger beam diameter used.

Changed Ion Beam Conditions
I_p = 465 nA

Specimen #75 - 0.10%

Fig 11b

6.80×10^{-4}
7.44×10^{-4}
7.22×10^{-4}
7.22×10^{-4}
7.21×10^{-4}
7.16×10^{-4}
7.25×10^{-4}
7.14×10^{-4}
7.22×10^{-4}
6.47×10^{-4}

$\sigma = .23$ (3%)

$\bar{A}v = 7.11 \times 10^{-4} \pm 0.2$
(vs 1.0×10^{-3} for I_p =

Specimen #584 - 0.001%

Fig 11b

1.624×10^{-5}
2.1×10^{-5}
2.02×10^{-5}
1.98×10^{-5}
2.06×10^{-5}

2.56×10^{-5}
1.89×10^{-5}
2.32×10^{-5}
2.19×10^{-5}
1.71×10^{-5}

$\sigma = .24$ (12%)

$Av = 2.05 \times 10^{-5}$ (vs 3.5×10^{-5} for I_p

A-4

Energy, Mines and Resources Canada
Research and Technology
Canada Centre for Mineral and Energy Technology,
555 Booth Street
Ottawa, Ontario
K1A 0G1

Énergie, Mines et Ressources Canada
Recherche et technologie
Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa (Ontario)
K1A 0G1

A-4

Your file Votre référence

Our file Notre référence

cheat: Silver in Sea [illegible]

February 21, 1983

Dr. N.S. McIntyre,
Technical Director,
Surface Science Western,
Centre for Interdisciplinary Studies
in Chemical Physics,
Faculty of Science,
University of Western Ontario,
London, Ontario
N6A 3K7

Dear Dr. McIntyre:

Many thanks for your letter dated February 15, which enclosed the results from your ion microscopic studies of our synthetic Ag-bearing PbS standards.

As you are probably aware, Louis is presently out of town, he should be back in the first week of March. I am sure that your results are going to be of great interest to him, it sure looks quite promising.

Sincerely yours,

Gilles Laflamme

J.H.Gilles Laflamme

JHGL/tj

Canada

A-5

D.S.S. (SILVER)

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario.
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

A-5

1983/09/07

Dr. Louis J. Cabri
Research and Technology
Canada Centre for Mineral
& Energy Technology
555 Booth Street
Ottawa, Ontario K1A 0G1

Dear Louis,

I believe that we have made enough progress (or perhaps recovered enough ground) that things look hopeful again. The attached results suggest that most of your PbS and ZnS reference standards do fall on a straight line. Furthermore our sensitivities for both silver and indium look good.

The sensitivities for the ZnS standards, at the moment, appear better than those for PbS. This is only because we found that we had to use an O^- beam for ZnS specimens because of their greater tendency to charge. Using O^-, we found, in fact, a lower background in the Ag^+ region. It should be quite straightforward to run all specimens with O^- in future and thus improve our data for the PbS specimens.

The indium concentrations were not run in this present work. That can be done quite simply when we rerun your PbS specimens using O^- to obtain better sensitivity. We note from the spectrum for specimen 604A that the sensitivity of In^+ is about tenfold greater than for silver (see attached spectrum).

The results for the ZnS standards are:

Specimen Number	% Ag	I^+_{Ag}/I^+_{Zn}
603	0.109	$3.1 \times 10^{-2} \pm 0.1$ (σ)
604	0.055	$2.1 \times 10^{-2} \pm 0.15$
606	0.027	$8.2 \times 10^{-3} \pm 0.5$
607	0.011	$2.1 \times 10^{-3} \pm 0.1$
605	-	$1.0 \times 10^{-4} \pm 0.1$

A-5 (recal)

The data plotted in Figure 1 shows a reasonably linear response (or a very good response if you want to ignore #603). I believe that the I^+_{Ag}/I^+_{Fe} + detection limit is $<4 \times 10^{-6}$ (equivalent to 0.5 ug/g). Therefore specimen 605 may have a trace silver concentration of 20 ug/g.

The data for PbS is given below:

Specimen Number	% Ag	I^+_{Ag}/I^+_n
575	0.10	4×10^{-3}
580	0.05	2.8×10^{-2}
581	0.025	$1.5_5 \times 10^{-2}$
582	0.01	4.8×10^{-3}
569	0	1×10^{-3}

The plot (Figure 2) is again fairly linear, except that there is one point (#575) that is completely out of range. It showed poor agreement in our previous trial as well and may have poor homogeneity. As I suggested earlier, we're not happy with the sensitivity shown in this series and will rerun them.

Homogeneity of these materials was checked by depth profiling and any change of ion ratio was detected. Only after the ratio became constant within the statistical variation was the data accepted.

The primary beam diameter used for all this work was 50 um. Rather than using a smaller beam in future we will reduce our field aperture to a 10 um area if required to do so due to particle size. The O^- beam stability is not affected by positioning it partly on the araldite and partly on the mineral.

It thus appears that this particular mounting material can be successfully used for silver and indium determinations. Our nextstep will be to reply to your request for proposal as rapidly as possible. We hope that, having rerun the PbS specimens as well as some of the supplied minerals, this could be written up for publication.

I look forward to seeing you on the 20th of September.

Yours very truly,

S Stewart

N.S. McIntyre
Project Manager
Surface Science Western

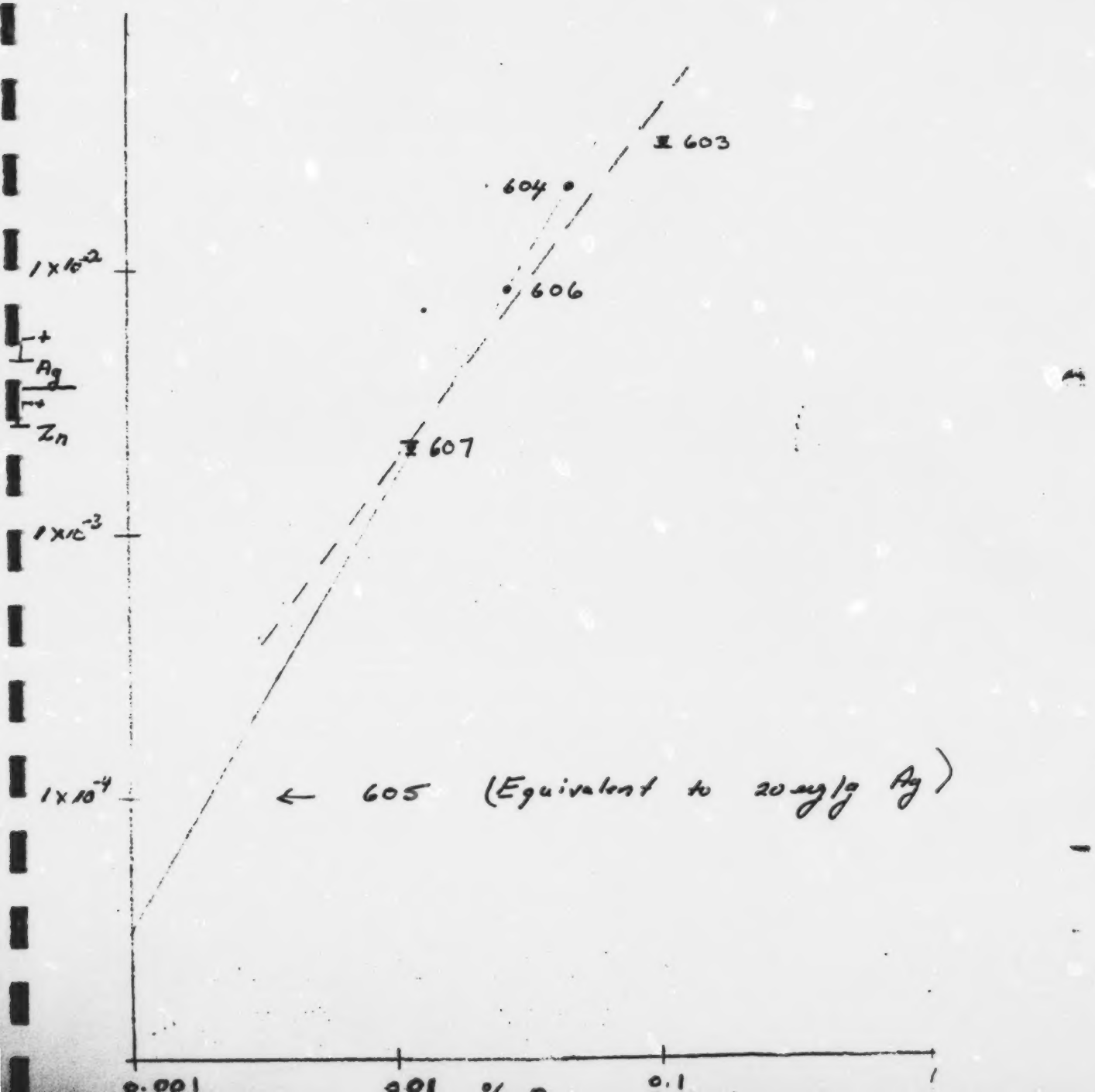
cc: W.J. Chauvin
D.S.S. File (Silver in Minerals)

A-5 (cont'd)

Figure 1

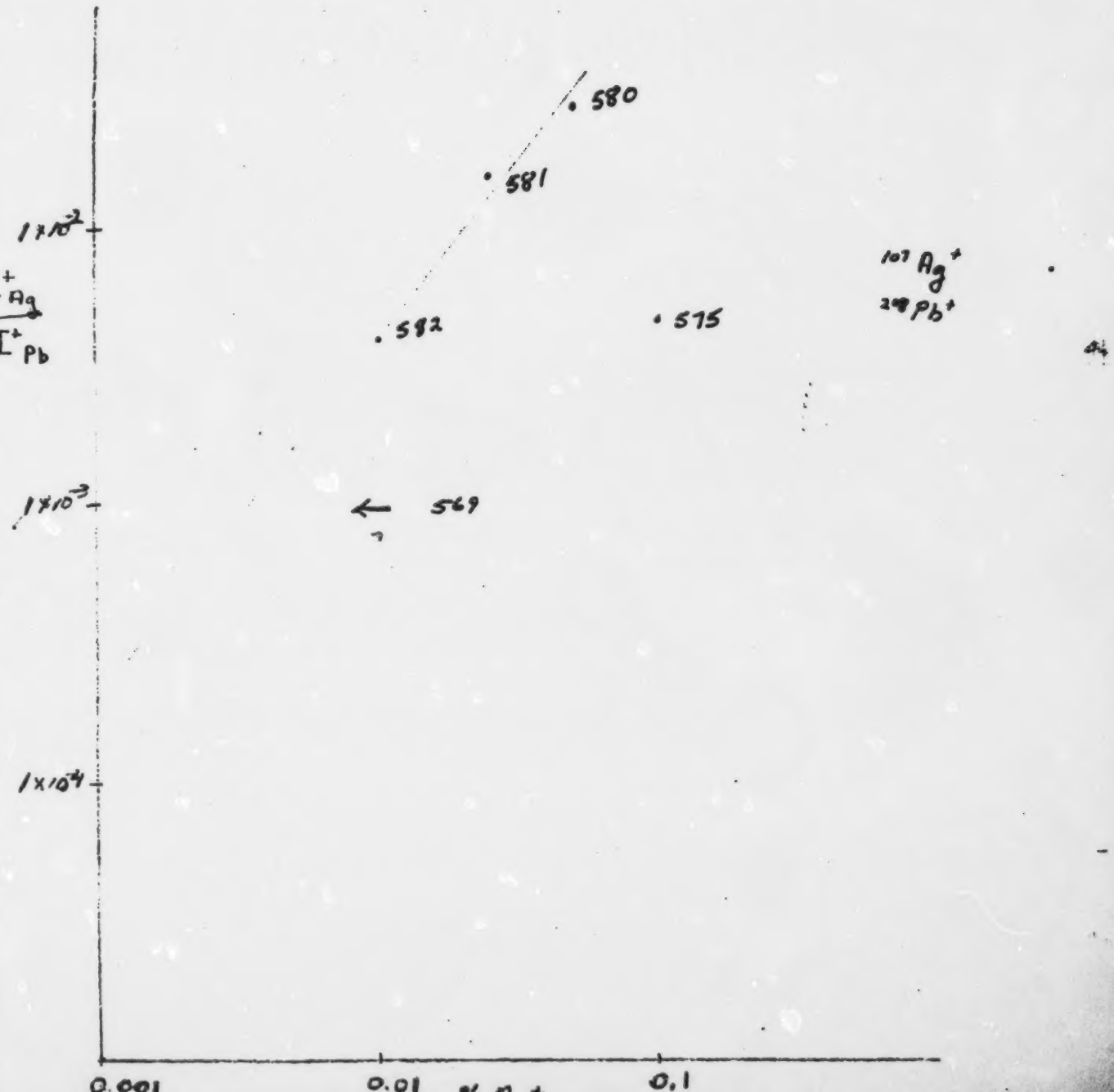
Ag Calibration Plots for Zn Fe S standards

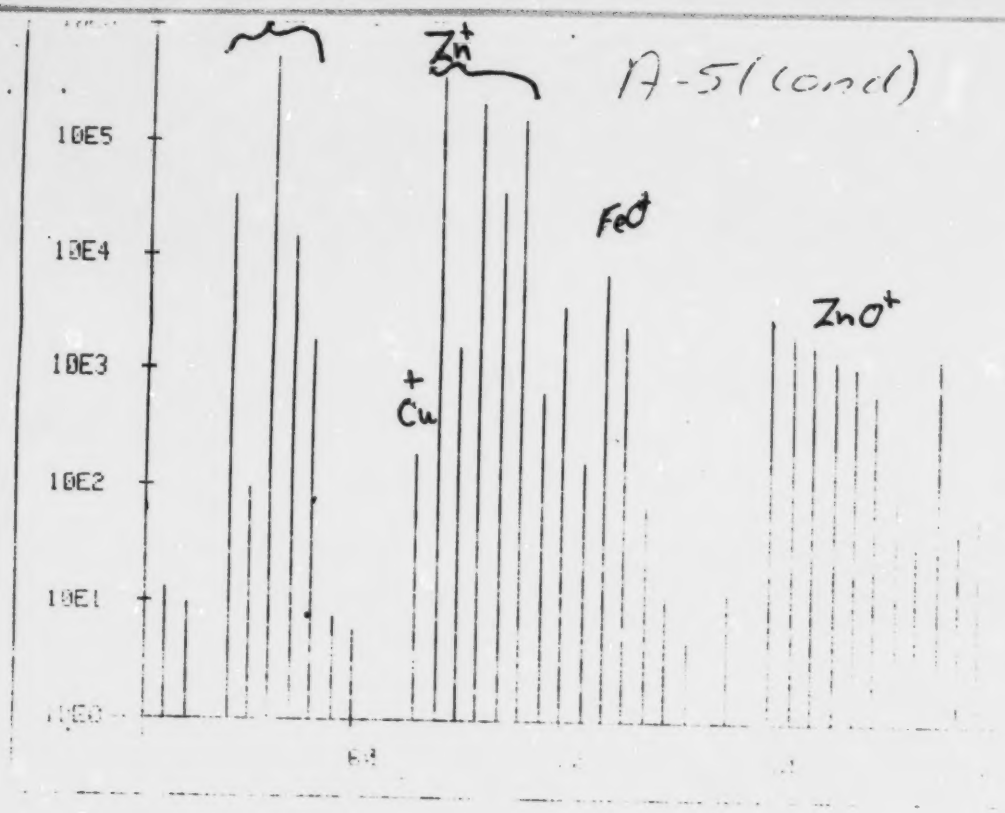
O^- primary beam -

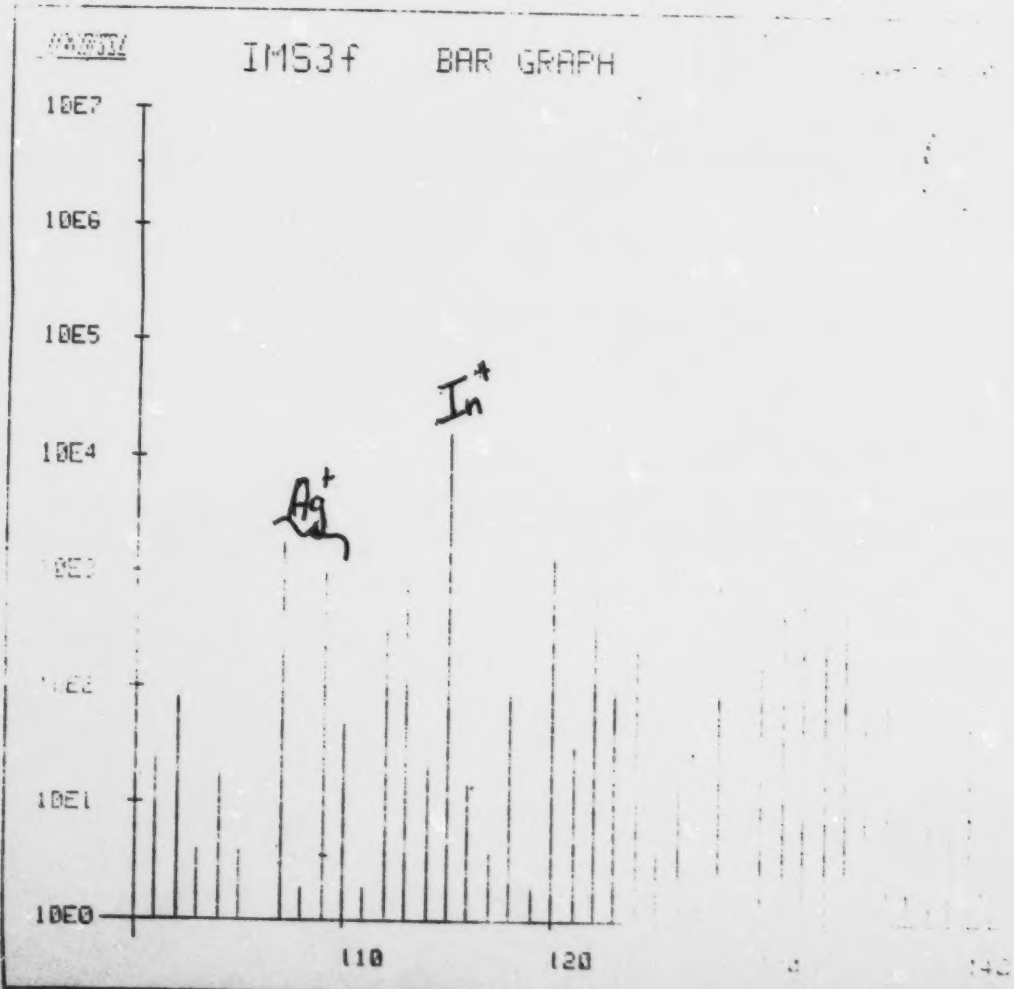


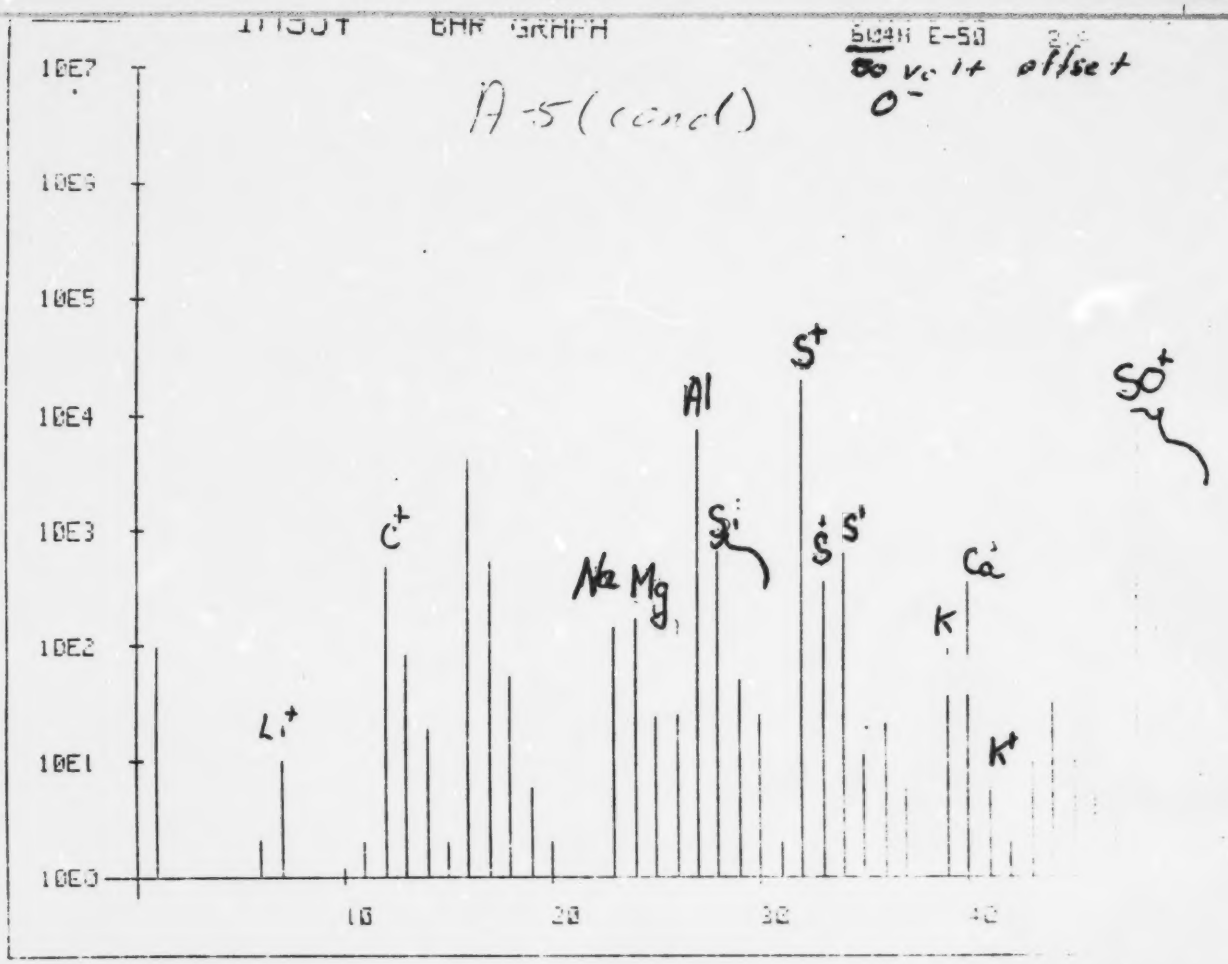
A-5 (cont)

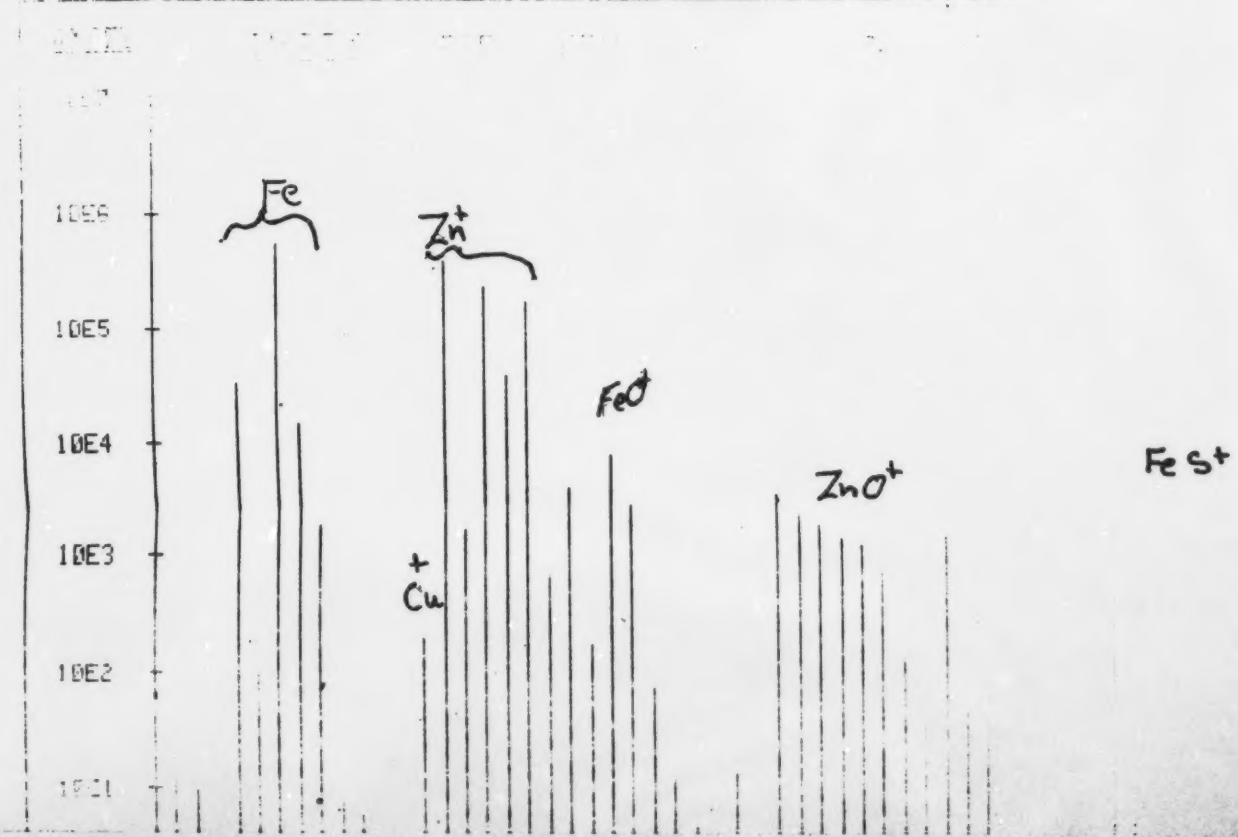
Figure 2
Ag calibration plot
for PbS standards.
O_2^+ primary beam.




A-5 (cont'd)
10E5
10E4
10E3
10E2
10E1
Zn+
FeO+
ZnO+
Fe S+
+
Cu


IM53f BAR GRAPH
10E7
10E6
10E5
10E4
10E1
10E0
In+
Ag+
110
120


BAR GRAPH
6024H E-50
80 volt offset
A-5 (cont)
O⁻
10E7
10E6
10E5
10E4
10E3
10E2
10E1
10E0
10
20
30
40
Li+
C+
Na
Mg
Al
Si
S+
S
S'
SO+
K
Ca
K+


10E6
10E5
10E4
10E3
10E2
Fe
Zn+
Cu+
FeO+
ZnO+
Fe S+

A-6

The University of Western Ontario

Centre for
Interdisciplinary Studies
in Chemical Physics
Faculty of Science
London, Canada
N6A 3K7

Telephone (519) 679-6371
Telex UWO LIB LDN 064 7134

August 30, 1983

Dr. Louis J. Cabri
Canada Centre for Mineral & Energy Technology
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis,

This is to acknowledge your letter of August 16th, proposing a joint project to determine if trace concentrations of silver can be quantitatively determined in galena and sphalerite matrices. I agree to the arrangements suggested and await the samples to be sent by Mr. Laflamme.

Each sample will be analyzed as follows:

(a) Complete bar graph mass spectra (to M/e = 250)
(b) Partial mass spectra (Spectra of mass regions:
50-70, 110-110 and 200-210)
(c) Line scans across sample surface measuring masses
32, 57, 63, 68, 107 and 109

I assume that the specimens will be mounted and prepared in the same manner as those submitted previously.

Yours very truly,

Stewart

N.S. McIntyre
Technical Director
Surface Science Western

cc: J.H.G. Laflamme (CANMET)
W. Chauvin (SSW)

B

EFFECTS OF PRIMARY BEAM PARAMETERS

(September 1983 - March 1984)

Contract File.
Silver in Sulphide Mineral.

Energy, Mines and Resources Canada
Research and Technology
Canada Centre for Mineral and Energy Technology,
555 Booth Street
Ottawa, Ontario
K1A 0G1

Énergie, Mines et Ressources Canada
Recherche et technologie
Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa (Ontario)
K1A 0G1

Your file Votre référence

Our file Notre référence

BY COURIER

B-1

September 28, 1983

Dr. N.S. McIntyre,
Technical Director,
Surface Science,
Western Centre for Interdisciplinary
Studies in Chemical Physics,
University of Western Ontario,
London, Ontario
N6A 3K7

Dear Stewart:

As we discussed at UWO on the 20th, I return the two polished sections for the test analyses. These sections have been remounted and repolished. The section with the natural minerals has ∿0.5 cm diameter circles (one or two each) which should help you to place the beam. I would be interested to see how close to the center can be achieved.

Looking forward to the results of a successful analyses on the 4th October.

Kindest regards.

Yours sincerely,

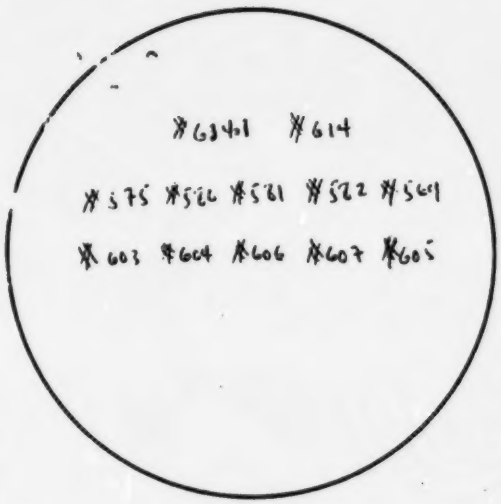
Louis J. Cabri

LJC/tj

Canada

B1-(cont'd) CANMET 23/8/83

Standard Blocks for Western Ontario Univ.



Block A.

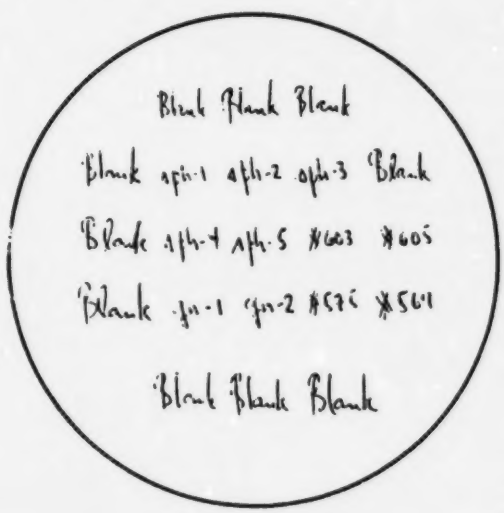
(Standards mounted in araldite)

	Cu	In	Ag	Fe	S
#634-1	29.88	0.160	0.115	36.91	32.94
#614	30.29	~~0~~	~~0~~	36.72	32.99

	Pb	Ag	S
#575	86.50	0.10	13.40
#580	86.55	0.05	13.40
#581	86.575	0.025	13.40
#582	86.59	0.01	13.40
#569	86.60	~~0~~	13.40

	Zn	Fe	Ag	In	S
#603	64.198	2.560	0.109	0.114	33.018
#604	64.317	2.541	0.055	0.057	33.030
#606	64.366	2.541	0.027	0.028	33.037
#607	64.384	2.552	0.011	0.011	33.041
#605	64.193	2.752	~~0~~	~~0~~	33.055

64



Block B.

(Specimens & Stubs mounted with CERAC Alloy in contact with the brass holder. Final impregnation with araldite for polishing.)

Sph-1 to sph-5 are natural sphalerites from Nanisivik (N.W.T.); gn-1 and gn-2 are natural galenas from the same locality.

- [redacted]

inclusion-free areas on natural sphalerite and galena circled ...

sph-1	two areas	gn-1	two areas
sph-2	two areas	gn-2	" " .
sph-3	one area		
sph-4	one area		
sph-5	one area.		

B-21

"Silver in Sulphides" File

B-2

Dr. L. J. Cabri,
Mineral Sciences Laboratories
CANMET
Canada Dept. of Energy Mines & Resources
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis;

Weve completed the three additional tasks of the silver/indium in zinc sulphide investigation:

1 Effects of field aperture & beam current on ion current ratios. These are given on section 1 of the attached Appendix. For silver (#60) there are only minor changes with beam size or field aperture, ~~I therefore think that such effects~~, while for indium one result is significantly different from the others. Also, note that ~~the difference in~~ each ratio is about ~~two-fold~~ a factor of four different from the results of a few days earlier, done with a different piece of #603. I conclude that ~~probably~~ probably the beam size & field aperture changes aren't significant. Some of the difference in the ratio measured on two different days could be due to a change in background setting up the calibration plot.

2. Analysis of unknown standards. Most silver

concentrations are well above the blank value, and, in fact one area (2b) has a very high spot concentration. Indium concentration dir, however, ~~lower tha~~ very consistent and are clearly lower than the "blank" (#605) which you supplied. I suspect ~~that~~ the, therefore, that your blank specimens have a finite concentration of indium and probably silver.

Finally, ~~ther~~ I've attached a line scan made between points 2(b) (high silver) and 2a (low silver) on sph 2. Except for the extreme left hand side, I don't believe the quantitative data. We've discovered that your holder track is misaligned, so that in a line scan the ion beam does not stay in focus. Nonetheless, the line scan qualitatively shows some silver and indium variations. Well try for a better one for the paper, after fixing our alignment problems.

No silver ion images could be detected in the mineral.

A copy of our proposal to D.S.S. is enclosed.

Best regards

yvt.

1-Hyperion 13-2 (cont'd).

Analysis of ZnS - Oct 6, 1983

① Effect of Analysis Area on Ag/Zn & In/Zn Ratio

	Specimen 13 Ag (0.1%)	In (0.1%)		Specimen 605 Ag (0.1%)	In (0.1%)
= 30 um ; F = 150	1.3×10^{-2}	5.2×10^{-1}	475	1.1×10^{-4}	4.9×10^{-4}
= 30 um ; F = 60	1.5×10^{-2}	6.2×10^{-1}	475	6.6×10^{-5}	4.3×10^{-4}
= 70 um F = 60 ↑ Rocked	1.2×10^{-2}	2.9×10^{-1}		6.8×10^{-5}	4.5×10^{-4}
Previous result *	2.1×10^{-2}	3.6×10^{-1}		8.2×10^{-5}	

* different specimen

② Analysis of Unknown Sphaelerites

For Calibration Graphs used values of:

Ag 0.11% 0 -1.3×10^{-2}

In 0.11% 5.2×10^{-1}

Ag 'blank' -1.1×10^{-4}

In 'blank' -4.9×10^{-4}

Unknown Sphalerite Values

	Silver Ratio	Ag Conc. [ug/g]	Indium Ratio	In Conc [ug/g]
Sph-5	9.4×10^{-4}	75 ± 10 (16)	3.5×10^{-4}	0.4 ± 0.2
Sph-4	1.1×10^{-3}	90 ± 10 (16)	2.5×10^{-4}	0.4 ± 0.2
Sph-3	1.55×10^{-3}	135 ± 10 (16)	2.5×10^{-4}	0.4 ± 0.2
Sph-2a	7.4×10^{-4}	58 ± 10 (16)	2.3×10^{-4}	0.4 ± 0.2
Sph-2b	4.3×10^{-2}	3500 ± 500 (16)	2.3×10^{-4}	0.4 ± 0.2
Sph-1a	5.0×10^{-4}	37 ± 10 (16)	2.5×10^{-4}	0.4 ± 0.2
Sph-1b	2.3×10^{-3}	180 ± 20 (16)	2.5×10^{-4}	0.4 ± 0.2

B-2 (cont'd)

A ZnS Analysis - Large O- Beam (1)

Specimen	Ag Concentration (%)	I^+Ag/I^+Zn	In Concentration (%)	I_{In^+}/IZn^+
603	0.109	$1.85\pm0.1[5] \times 10^{-2}$	0.114	$3.85\pm 0.05[5] \times 10^{-1}$
604	0.055	$8.7 \pm 0.3[3] \times 10^{-3}$	0.057	$2.2\pm0.1[4] \times 10^{-1}$
606	0.027	$4.7\pm 0.4[3] \times 10^{-3}$	0.028	$1.20\pm0.05[3] \times 10^{-1}$
607	0.011	$1.78\pm0.05[3] \times 10^{-3}$	0.011	$4.3\pm0.1[3]_2 \times 10^{-2}$
605	0	$8.6 \pm 1[3] \times 10^{-5}$	0	$4.7\pm0.1[3] \times 10^{-4}$

(1) Ip = 40 nA, FA = 150 um

B ZnS Analysis - Small Analysis Area (2)

604	0.055	$2.78 \pm 0.2[2] \times 10^{-3}$	0.057	$8.4 \pm 0.1(2) \times 10^{-2}$
606	0.027	$1.1 \pm 0.05[3] \times 10^{-3}$	0.028	$3.0\pm0.1[3]_2 \times 10^{-2}$
607	0.011	9.3 ± 1 [3] $\times 10^{-5}$	0.011	$1.9 \pm 0.3[3] \times 10^{-2}$

(2) Ip = 140 nA FA = 3 CA = 3 (FA = 8 um)

(see Fig. 1)

A PbS Analysis - Large Area, Low Current (1)
(Ser I)

Specimen	Ag Concentration %	$I_{Ag}/I_{Pb}(208)$
575	0.10	$2.1 \pm 0.1 \times 10^{-4}$ [2]
580	0.05	$3.35 \pm 0.15 \times 10^{-4}$ [4]
581	0.025	$2.75 \pm 0.1 \times 10^{-4}$ [3]
582	0.01	$1.2 \pm 0.1 \times 10^{-4}$ [3]
569	0	$9.0 \pm 1 \times 10^{-6}$ [3]

(1) - 30 nA, 150 um

B PbS Analysis - Large Area, Higher Current (2)
(Ser II)

575	0.10	$2.2 \pm 0.05 \times 10^{-4}$ [4]*
580	0.05	$3.47 \pm 0.05 \times 10^{-4}$ [3]
581	0.025	$3.0 \pm 0.1 \times 10^{-4}$
582	0.01	$7.5 \pm 1.5 \times 10^{-5}$
569	0	4×10^{-6}

(2) 60 nA, 150 um

* Corrected isotope ratio to Pb-208
(See Fig. 2)

C PbS Analysis - Large Area, Lower Current
(Ser III) - Different Specimens Areas)

580	0.05	$4.1 \pm 0.2 \times 10^{-4}$
581	0.025	$2.9 \pm 0.2 \times 10^{-4}$
582	0.01	$9.3 \pm 1 \times 10^{-5}$
569 (different specimen)	0	$1.0 \pm 0.2 \times 10^{-5}$

D

Unknown Specimens	Calculated Concentration	
UGA1(Area 1)	0.0027± 0.0000 (27 ± 8 ppm)	$2.4 \pm 0.5 \times 10^{-5}$
UGA1(Area 2)	0.0090±0.002 (90 ± 20 ppm)	$8.3 \pm 2 \times 10^{-5}$
UGA2(Area 1)	0.0023±0.0009 (23 ± 9 ppm)	$2.0 \pm 0.5 \times 10^{-5}$
UGA2(Area 2)	0.0020±0.0004 (20 ± 4 ppm)	$1.8 \pm 0.2 \times 10^{-5}$

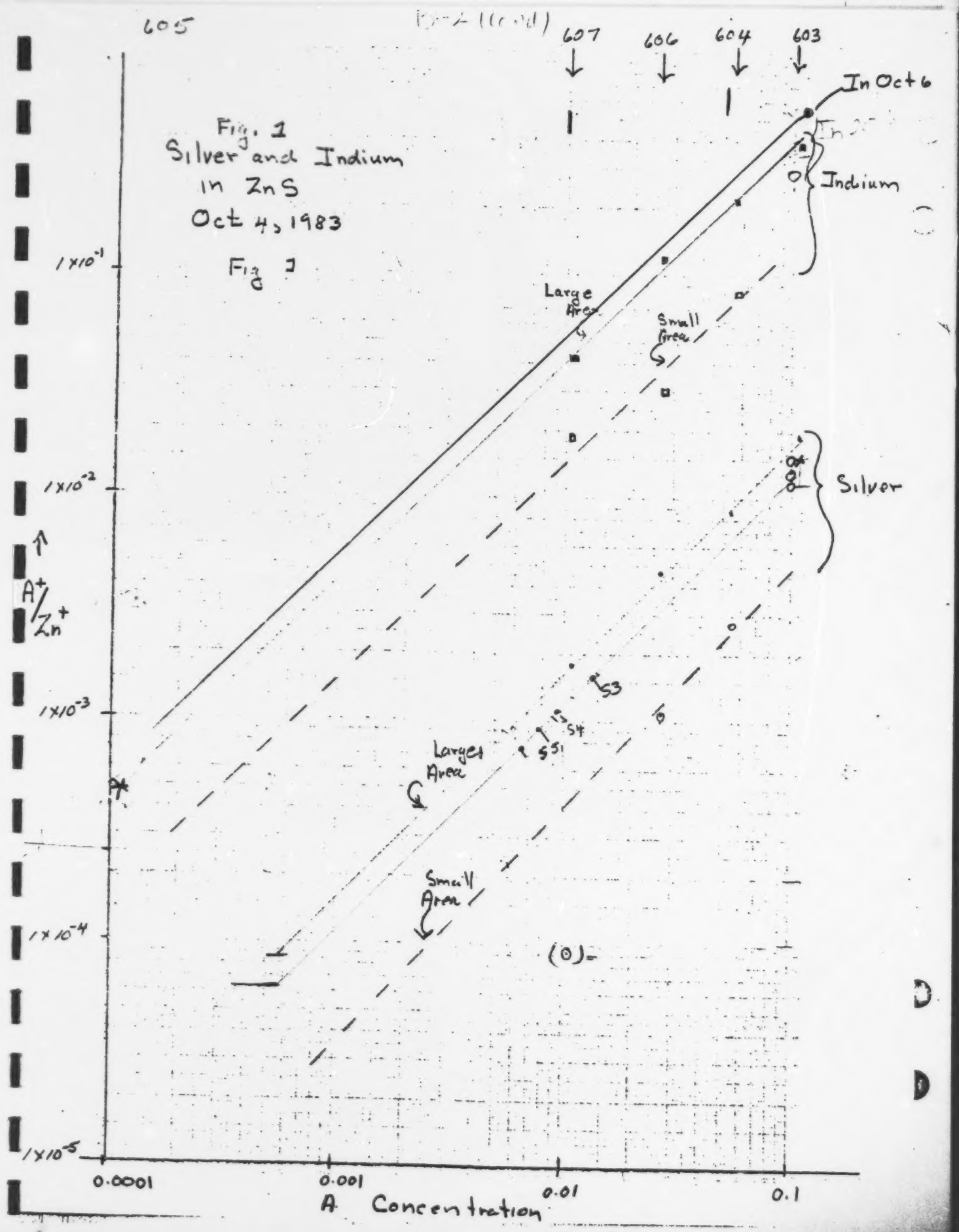
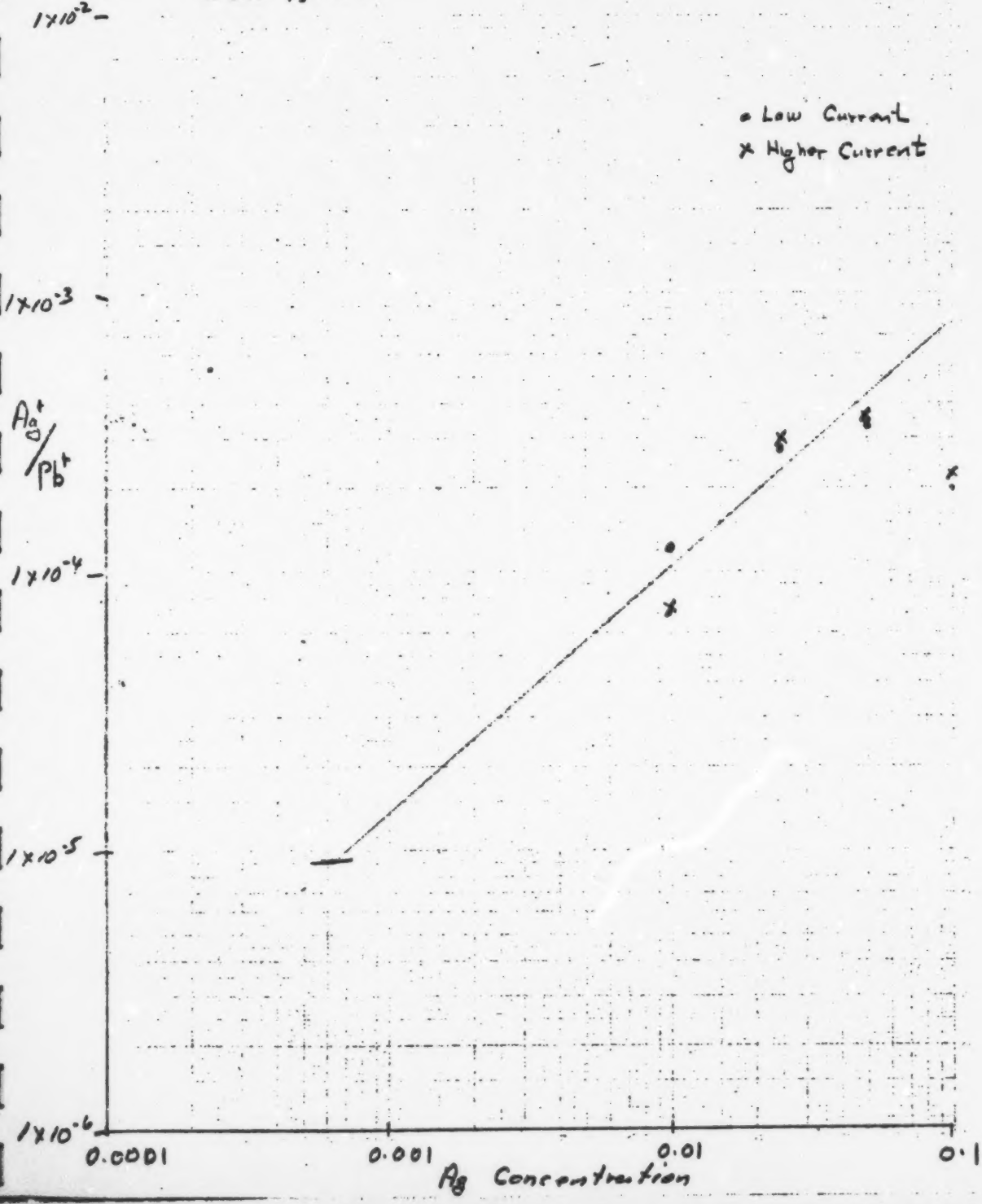


Fig. 1
Silver and Indium
in ZnS
Oct 4, 1983

Fig 1

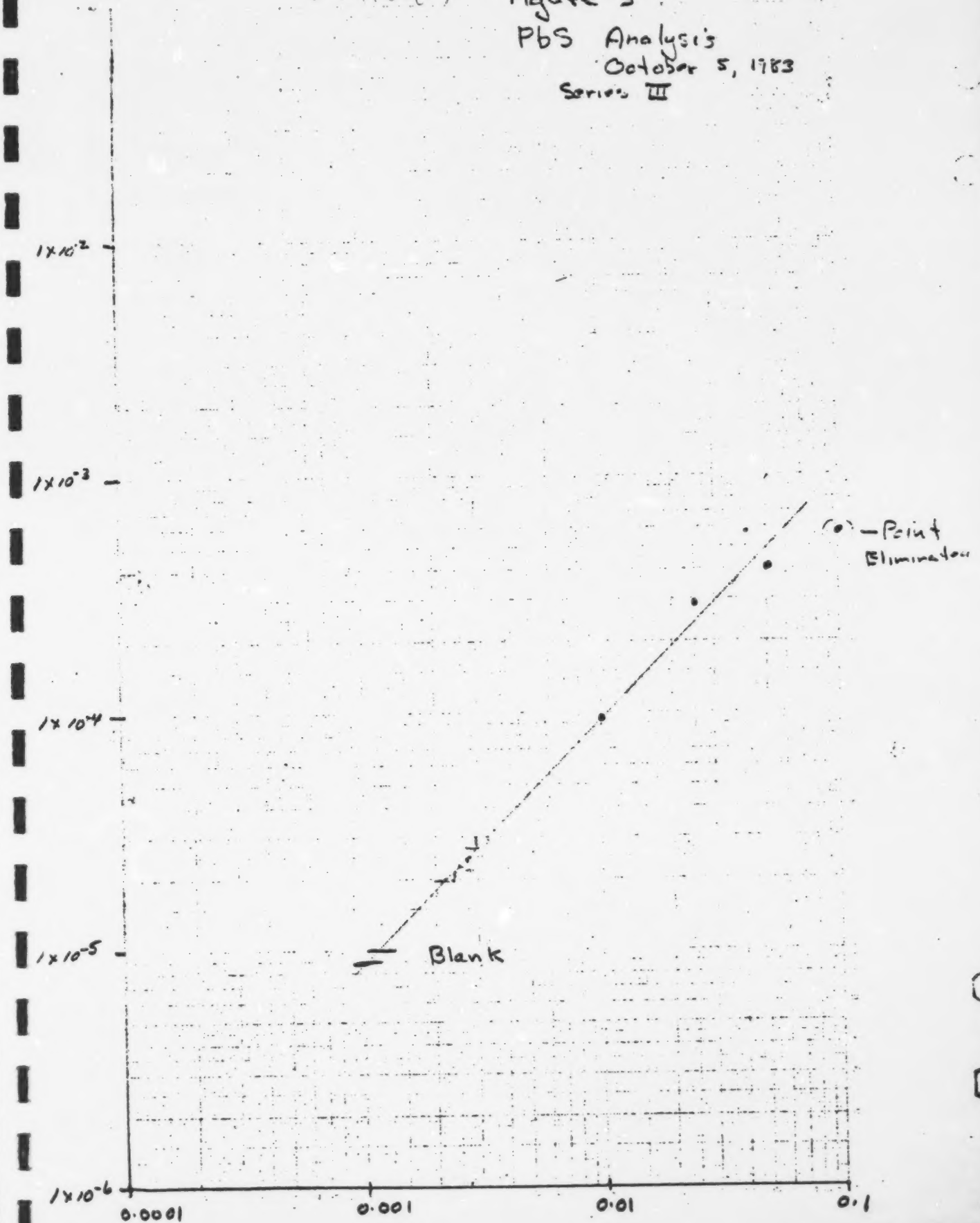
B-2 (cont'd)

Fig 2
Silver in PbS
Oct 4, 1983.



B-21(cont)

Figure 3.
PbS Analysis
October 5, 1983
Series III



B-3

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

Research and Technology
Recherche et technologie

Canada Centre for Mineral and Energy Technology,
555 Booth Street
Ottawa, Ontario
K1A 0G1

Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa (Ontario)
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Your file Votre référence

Our file Notre référence

BY COURIER

B-3

October 27, 1983

Dr. N.S. McIntyre,
Technical Director,
Surface Science,
Western Centre for Interdisciplinary
Studies in Chemical Physics,
University of Western Ontario,
London, Ontario
N6A 3K7

Dear Dr. McIntyre:

As promised by Dr. Cabri earlier this week in his letter, please find enclosed two additional polished sections labelled "A" and "B" containing various Ag and In-free standards. Different polishing techniques were used for these two blocks.

We hope you can run these specimens as soon as possible.

Sincerely yours,

J.H.G. Laflamme

Encl.
JHGL/tj

Canada

B-4

FILE: CABRI CORRES.
SILVER

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

B-4

1984/03/23

Dr. L.J. Cabri
Canada Centre for Mineral and Energy Technology
Energy Mines & Resources Canada
555 Booth Street
Ottawa, Ontario
K1A OG1

Dear Louis,

Please find enclosed a report of our work on block WA containing 38 sphalerite specimens. Because the length of time required to analyze each specimen with the smaller 10-20 um beam, is about twice that for the 30-40 um spot used in our earlier tests, we would like to adjust the billing accordingly. Our actual allocation of time spent during a typical day is as follows:

8:30 - 9:00 Instrument optimization
9:00 - 11:00 Calibration curve generation, plotting, and verification
11:00 - 17:30 Analysis of unknowns

During the actual analysis period a total of 20-24 analyses can be made. Based on an average of 22, the cost per analysis is $51 ($1125 per day) since the original cost estimated was $26, we wish to adjust for the difference by billing you for twice the number of specimens. Thus, in the present case, we will bill you shortly for 156x2 = 312 analyses. In addition, we will be billing for two SIMS development days (one in November and one in January) and for the supporting personnel time for those days.

This works out to be:

I Analysis: 312 analyses x $26	=	$8112
II Instrument Charge 2 days x 1000	=	2000
III Salaries:	=	600
TOTAL:		$10,712

The slides are included and the new ones will be along shortly.

Best regards.

Yours very truly,

N.S. McIntyre
Director
Surface Science Western

cc: W.J. Chauvin

B-5

SIMS Determination of Trace Concentrations of Silver and Indium in Sphalerites (Lot WA)

Interim Report

to

Canada Centre for Mineral & Energy Technology
Canada Department of Energy Mines & Resources
DSS Contract No. OST 83-00220

Surface Science Western
Faculty of Science
University of Western Ontario
London, Ontario

March 1984

13-5 (cont'd

SIMS Determination of Trace Silver and Indium in Sphalerite (Lot WA)

This report details the results of the SIMS analysis of 38 sphalerite [(Zn,Fe)S] specimens submitted to the Laboratory imbedded in a single block of Araldite. The block surface was coated with ~100 nm of gold prior to SIMS analysis. For analysis in the Cameca IMS-3F, an 8 keV, 1-2 na. beam of O^- ions was used, with a beam diameter of 10-20 um width. With such a relatively low ion beam current beam, penetration rates of the gold coating, and the damaged outer surface of the sphalerite, was slow (~10 minutes). The intensity of the $^{66}Zn^+$ peak was monitored until it reached a constant value, and then analyses were begun. These procedures resulted in considerably longer analysis times than was previously estimated.

For each standard or unknown, the intensities of $^{66}Zn^+$, $^{107}Ag^+$ and $^{115}In^+$, were monitored for a period of ten seconds* for each measurement. Five separate measurements were taken in sequence, in order to assess the precision of the measurement. Count rates for silver and indium isotopes were then ratioed to that of zinc. In Figure 1 a typical printout for a synthetic standard (32-A) is shown. It can be seen that there is no significant evolution in the Ag/Zn or In/Zn ratio over the period of five measurements. Moreover, the precision of the data taken during the five measurements is quite tight. For 32-A (270 ug/g Ag) the standard deviation of the five measurements averaged 1.4%. This translates to a 2σ(95% confidence level) precision of ± 8 ug/g on the 270 ug/g standard.

Reproducibility of a standard from one daily analysis session to the next is

* Ten seconds of measurement corresponds to about twenty seconds of real time.

also very important. The Ag/Zn ratio obtained over a series of sessions for specimen 32 (A or B) is shown below:

Analysis Data	Ratio Obtained
84/01/24 (A)	3.85×10^{-2}
84/02/03 (A)	1.81×10^{-2}
84/02/03 (B)	6.81×10^{-3}
84/02/08 (A)	1.34×10^{-2}
84/02/10 (A)	8.26×10^{-3}
84/02/17 (A)	7.46×10^{-3}
84/02/24 (A)	7.76×10^{-3}
84/03/03	7.36×10^{-3}
84/03/12	7.80×10^{-3}

Taken over the last five results, the average is $7.27 \times 10^{-3} \pm 0.30 \times 10^{-3}$. This is equivalent to a 2σ precision of ± 21 ug/g for the 270 ug/g standard

It appears that the standard has gradually decreased somewhat in value over a number of sessions and has, moreover, been analyzed with better and better precision over the last few sessions. This is probably due to a more homogeneous region of the standard having been reached as a result of sputter penetration into the interior. The top surface of the standards may be contaminated during polishing, to a greater extent than was previously anticipated.

Figure 2 shows calibration plots for areas A & B in the respective standards. The line drawn for both elements represents the average slope of 7 different plots, most of which are not shown on the graph. For indium, both areas give values which fall close to each other, as well as close to the average for all runs.

B-5 (cont'd)

Even the low indium standard, (17 ug/g) gives ratios lying close to the average line. For silver, however, it appears that the lowest standard lies wellabove the curve, and even the 55 ug/g silver standard shows considerable variation in its value. This is believed to result from a high silver blank value in the standards. Specimen 36, a synthetic blank, was determined to contain ~25 ug/g silver. Thus the lowest silver standard (16 ug/g) and the blank were not used further.

Figure 3 shows the range of values for standards and the average fit of the data.For each daily session a separate set of data was obtained and the least squares slope and intercept were obtained (see Figure 4).

If the unknown values were determined using uniquely the calibration curve established for the session ,the analytical precision of the resulting data can be calculated fairly accurately. The standard deviations of the slope and intercept can be determined from the least squares calculation (1). Given below are the precisions calculated for two separate sessions with a different number of points in the calibration plot.

Case 1 - 1984/02/17 (five point graph)

$y = 43357 \pm 1100x^{*} - 32.9 \pm 15$

Concentration ug/g	Precision (2σ) ug/g	% Precision (2σ)
1090	66	6
270	36	13
20	30	>100%

B-3(cont)

Case 2 - 1984/02/24 (three point graph)

$y = 31648 \pm 1300\ X + 3.5 \pm 37$

Concentration ug/g	Precision (2σ) ug/g	% Precision (2σ)
1090	116	11
270	76	28

The precision above is associated with the calibration graph only. To this, must be added the estimated 8% (2σ) precision in making the unknown measurement . The precisions given for measurements at the 20 ug/g level are indeed large. However, it must be recalled that a good deal of corroborating data on theslope and intercept of the analytical curve has been accumulated as a result of successive analyses (see Figure 2). Thus the figures here represent an extreme boundary condition. The other extreme would be to use all points obtained to date in the least squares fit.

For each session, a calibration curve was established and it was verified that it lay within error limits of that for the preceding day. Slopes determined for several of silver calibration graphs are given below:

Session	Slope
84/02/08	37800
84/02/17	34030
84/02/24	31650
84/03/05	39360

During subsequent analysis of unknowns, periodic reference checks of a calibration standard were made.

(5-Second)

The data obtained for the unknowns is given in Table 1, while the original reference microphotograph is included as Figure 5. Early in the measurement of the unknowns, it became apparent that most specimens exhibited a negative gradiant of silver concentrations during the 3 minute analysis. Indium also exhibited this property in certain specimens, but generally to a lesser extent than for the silver. Silver concentration, in some cases, dropped to less that half of the original value. The intensity ratio values for two typical specimens are given below:

Time	Specimen 8-I-1 Ag/Zn	Specimen 3-A-2 Ag/Zn
2 mm	2.08×10^{-2} (861 ug/g)	1.08×10^{-2} (406 ug/g)
4 mm	1.88×10^{-2}	8.62×10^{-3}
6 mm	1.72×10^{-2}	6.83×10^{-3}
8 mm	1.62×10^{-2}	5.74×10^{-3}
10 mm	1.56×10^{-2} (628 ug/g)	4.95×10^{-3} (176 ug/g)

In each case where an evolution of values is noted, the range is reported in Table 1, rather than an average. It is conjectured that silver in these natural specimens is affected by the heat of polishing, to a much greater extent that is the silver (or indium) in the synthetic standards.

(1) Kennedy, J.B. and Neville, A.M. "Basic Statistical Methods", Harper, New York, 1976.

Table 1

RESULTS - SPECIMEN WA

Specimen	In µg/g	Ag µg/g
1-A-1	63-59	74-16
1-B-1	94-89	368-135
2-A-1	117-98	228-74
2-B-1	119-95	394-183
3-A-1	76-63	279-159
3-A-2	93-68	406-176
3-B-1	71-58	355-168
3-B-2	76-60	369-175
4-A-1	98-68	557-210
4-A-2	89-66	407-173
4-B-1	88-66	350-167
4-B-2	63-61	141-101
4-C-1	73-63	244-155
4-C-2	84-63	380-192
5-A-1	105-80	399-182
5-B-1	88-70	253-129
5-C-1	112-91	343-154
6-A-1	27-23	190-115
6-A-2	34-25	275-135
6-B-1	24	143-111
6-B-2	30-22	246-128
6-C-1	30-23	250-136
6-C-2	30-22	227-121
7-A-1	45-30	257-117
7-A-2	40-28	241-98
7-A-3	39-26	199-87
8-A-1	63-56	10643-8618
8-A-2	14-11	1505-1080
8-B-1	13-11	1265-1002
8-B-2	13-10	1171-767
8-C-1	11-8	880-544
8-C-2	12-8	1009-567
8-D-1	12-8	758-396
8-D-2	9-6	1300-895
8-E-1	9-7	1006-761
8-E-2	11-7	1565-1019
8-F-1	10-7	701-327
8-F-2	10-6	780-450
8-G-1	7-5	1100-857
8-G-2	9-6	1038-667
8-H-1	9-8	1018-820
8-H-2	8-6	758-541
8-I-1	9-7	861-628
8-I-2	11-7	1130-674
8-J-1	14-10	359-195
8-J-2	14-9	568-350
8-K-1	11-7	1240-815
8-K-2	9-7	1039-708
8-L-1	9-6	817-605
8-L-2	9-7	808-569
8-M-1	9-6	966-666
8-M-2	9-6	921-595
8-N-1	8-6	1031-820
8-N-2	9-6	1015-711
8-O-1	7-6	1162-1037
8-O-2	7-6	583-456
8-P-1	15-11	577-412
8-P-2	13-10	921-662
8-Q-1	8-7	713-694
8-Q-2	8-7	927-819
8-R-1	8-6	793-761
8-R-2	7-6	755-712
9-C-1	236-174	352-153
9-D-1	101-70	413-172
9-E-1	114-79	366-154
9-F-1	152-106	333-118
9-G-1	9-6	246-140
9-H-1	12	114-85
9-I-1	<10	440-157
9-J-1	<10	408-180
9-K-1	28-17	286-99
9-A-1	203-215	75-61
9-B-1	91-96	93-73
10-A-1	73-65	105-71
10-B-1	62-61	125-95
10-C-1	64	53-45
10-D-1	63-62	91-65
10-E-1	62-63	87-68
10-F-1	63-65	76-65
11-A-1	47-45	115-67
11-B-1	46	104-69
11-C-1	46-47	88-64
11-D-1	46-45	68-55
12-A-1	149-155	96-69
12-B-1	152	82-59
12-C-1	166-170	95-72
12-D-1	154-151	105-76
13-A-1	116-121	96-74
13-B-1	111-113	89-74
13-C-1	115	386-111
13-C-2	114-112	103-75
13-D-1	140-122	359-171
13-E-1	198-151	259-76
13-F-1	175-147	360-113
14-A-1	11→ 10	124→ 87
14-B-1	10→ 9	103→ 81
14-C-1	10→ 10(10)	121→ 97
14-E-1	10→ 10	87→ 68
14-D-1	10	98→ 62
15-A-1	25→ 24	70→ 50
15-B-1	27→ 23	105→ 48
15-C-1	26→ 24	88→ 49
15-D-1	26→ 23	81→ 44
15-E-1	24	69→ 52
16A-1	286→ 269	110→ 66
16-B-1	283→ 243	126→ 74
17-A-1	229→ 224	74→ 55
17-B-1	250→ 244	98→ 73
17-C-1	309→ 234	186→ 75
18-A-1	941→ 947	117→ 99
19-A-2	105→ 92	190→ 106
19-B-1	84→ 76	92→ 45
19-C-1	81→ 76	80→ 47
19-D-1	72→ 69	60→ 40

Table 1 - Page 2

RESULTS - SPECIMEN WA (Page 2)

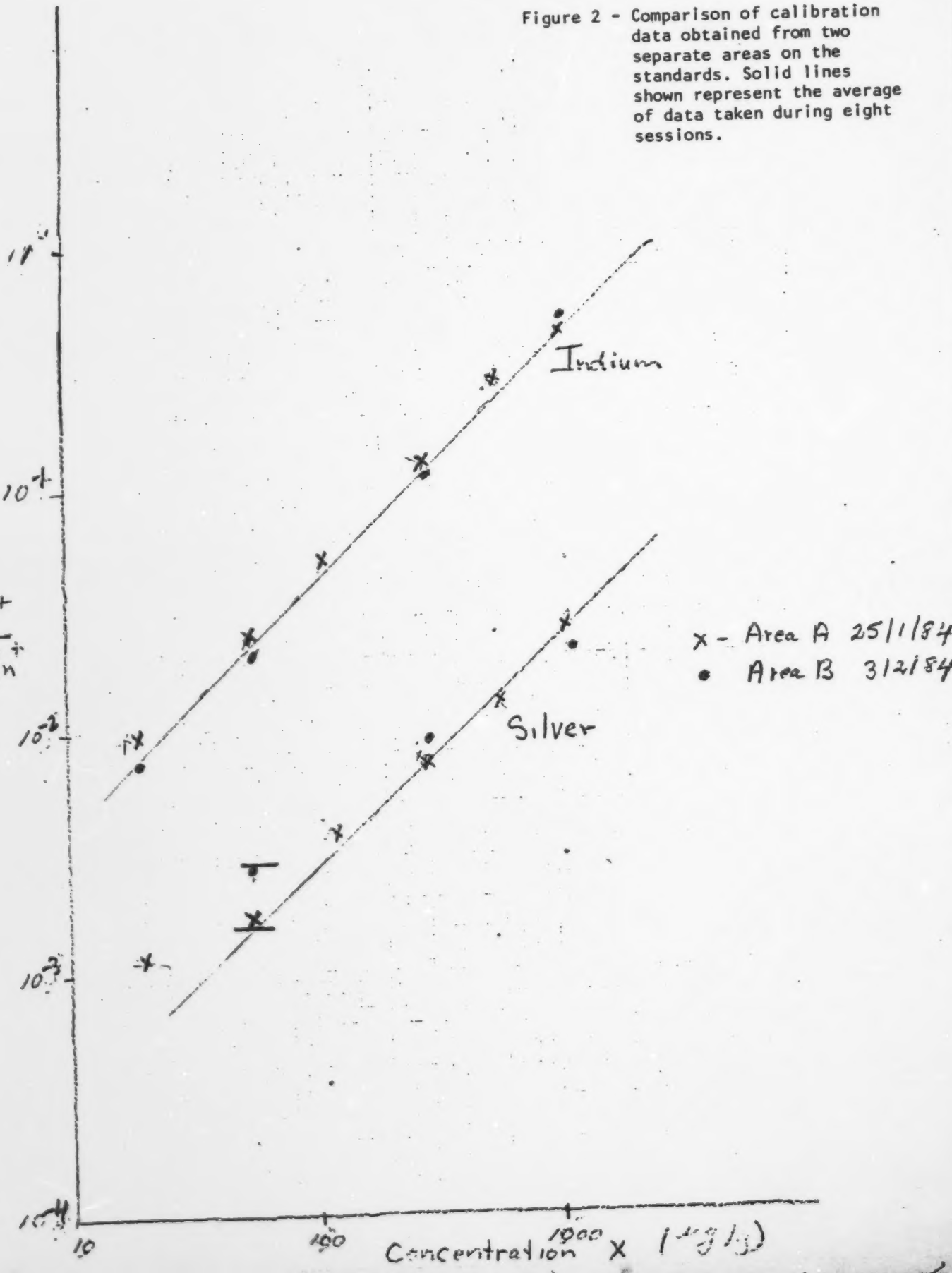
Specimen	In µg/g	Ag µg/g
20-A-1	16-14	331-213
20-B-1	11-10	181-97
21-A-1	816→ 722	118-72
21-B-1	874-800	127-77
21-C-1	907-766	164-90
22-A-1	1967-1680	183-106
23-A-1	12-10	151-93
23-B-1	13-11	214-114
23-C-1	12-10	135-88
23-D-1	11-10	123-89
24-A-1	120	70-45
24-B-1	127	45-33
24-C-1	128	92-68
24-D-1	125	50-40
24-E-1	74	79-51
25-A-1	35	80-47
25-A-2	34	59-43
25-A-3	33	59-44
26-A-1	187	109-81
26-B-1	202	82-50
26-C-1	181	56-41
26-D-1	210	62-40
26-E-1	200	46-26
27-A-1	8	68-51
27-B-1	8	82-49
27-C-1	8	86-54
28-A-1	1088	1090
28-B-1	1136	1047
28-C-1	955	802
28-D-1	909	775
29-A-1	536	496-536
29-B-1	480	456
29-C-1	472	472
30-36	Standards	
37-A-1	*84/01/26 1369	646-796
37-A-2	1291	1517-1658
37-B-1	1415	1559-1693
37-C-1	1217	1408-1490
37-D-1	1243	1458-1570
38-A-1	*84/01/26 7	85-53
38-A-2	7	35
38-B-1	7	37

(cont'd)

Figure 1 - Output data from the Cameca IMS-3F for specimen 32-A-10
(270 ug/g Ag, 280 ug/g In)

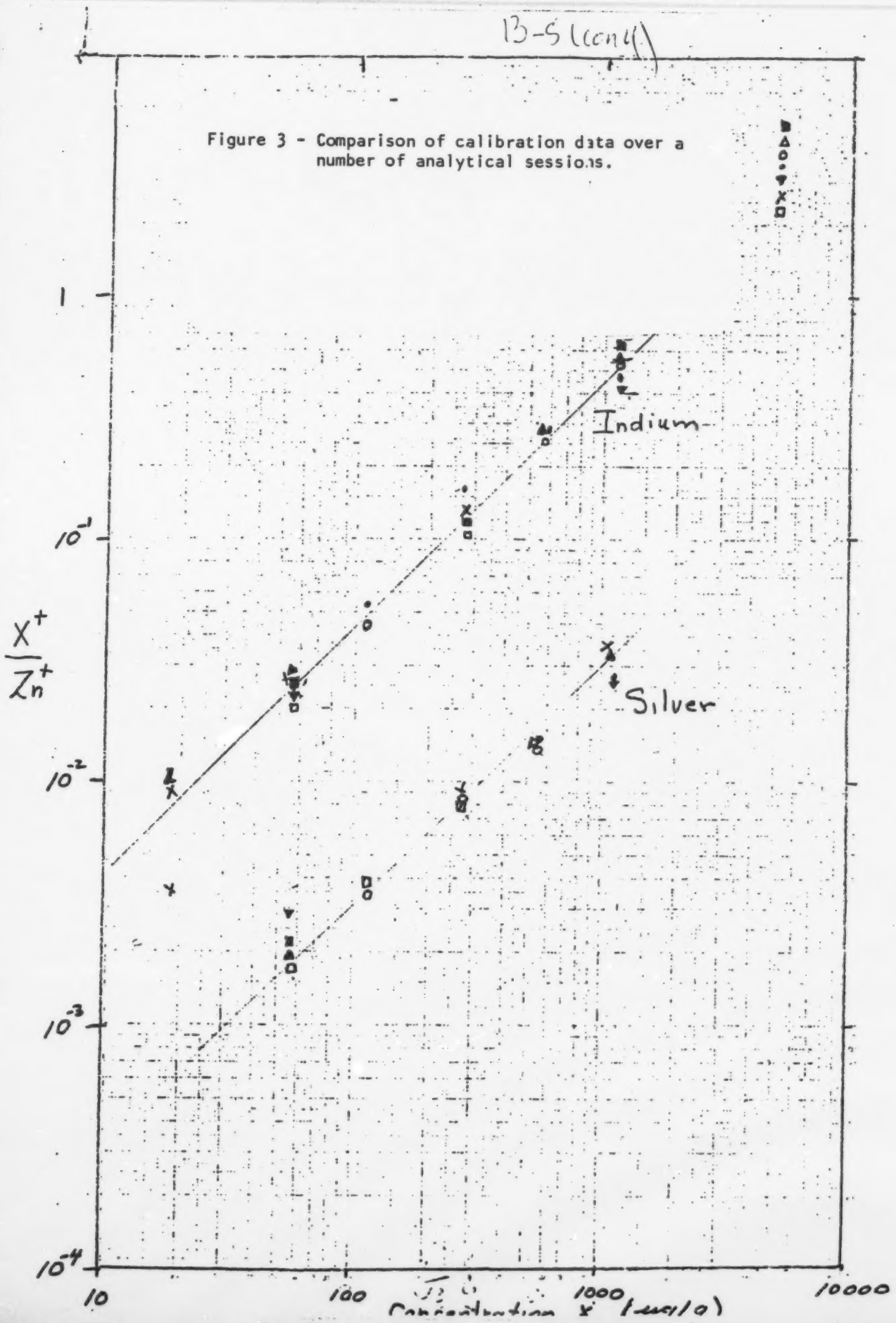
```
                    MEASUREMENT CONDITIONS
      MASS:                66
      MASS:               107
      MASS:               115

      REFERENCE MASS:      66
      MEASUREMENT TIME:    10
      WAITING TIME :        0
      NUMBER OF CYCLES:     5
      NUMBER OF BLOCKS:     1

                          RESULTS

                          BLOCK No:    1

                          CYCLE No:  1

       66/REF.-->        +1.000E+00          CNTS-->2.0406E+06
      107/REF.-->        +7.806E-03          CNTS-->1.5929E+04
      115/REF.-->        +8.280E-02          CNTS-->1.6897E+05
                                             BKGD-->0.000E+00

                          CYCLE No:  2

       66/REF.-->        +1.000E+00          CNTS-->1.6525E+06
      107/REF.-->        +7.889E-03          CNTS-->1.3037E+04
      115/REF.-->        +1.037E-01          CNTS-->1.7133E+05
                                             BKGD-->0.000E+00

                          CYCLE No:  3

       66/REF.-->        +1.000E+00          CNTS-->1.5634E+06
      107/REF.-->        +7.792E-03          CNTS-->1.2182E+04
      115/REF.-->        +1.102E-01          CNTS-->1.7229E+05
                                             BKGD-->0.000E+00

                          CYCLE No:  4

       66/REF.-->        +1.000E+00          CNTS-->1.5093E+06
      107/REF.-->        +7.827E-03          CNTS-->1.1814E+04
      115/REF.-->        +1.124E-01          CNTS-->1.6969E+05
                                             BKGD-->0.000E+00

                          CYCLE No:  5

       66/REF.-->        +1.000E+00          CNTS-->1.4377E+06
      107/REF.-->        +7.879E-03          CNTS-->1.1328E+04
      115/REF.-->        +1.156E-01          CNTS-->1.6618E+05
                                             BKGD-->0.000E+00
                          MEAN RATIO
      REFERENCE PEAK MASS:   66

      MASS: 66       MEAN:   +1.000E+00     STD DEV :   +0.000E+00
      MASS:107       MEAN:   +7.839E-03     STD DEV :   +4.350E-05
      MASS:115       MEAN:   +1.049E-01     STD DEV :   +1.312E-02

                          ATOMIC  o/o
      MASS: 66     PER CENT   :  +89.865
      MASS:107     PER CENT   :    +.784
      MASS:115     PER CENT   :   +9.430

                          WEIGHT (o/o)

      MASS: 66     PER CENT   : +83.643
      MASS:107     PER CENT   :  +1.063
      MASS:115     PER CENT   : +15.294
```

Figure 2 - Comparison of calibration data obtained from two separate areas on the standards. Solid lines shown represent the average of data taken during eight sessions.



B-5 (cont'd)

Figure 3 - Comparison of calibration data over a number of analytical sessions.



B-5 (cont'd)

Figure 4 - A typical least squares calibration plot.

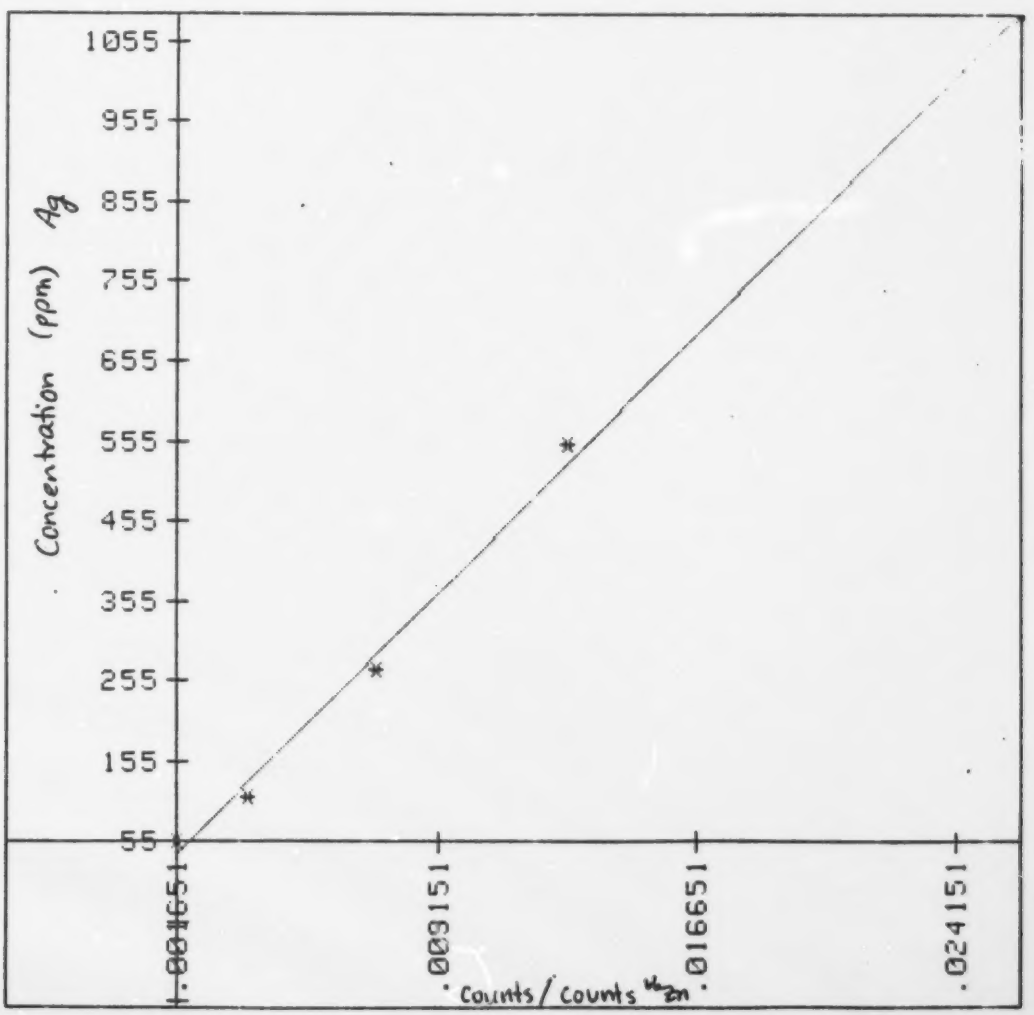
DATA

Point #1:	X=.001651	Y=55
Point #2:	X=.0036987	Y=110
Point #3:	X=.0073721	Y=270
Point #4:	X=.01292	Y=550
Point #5:	X=.02602	Y=1090

Linear model: Y=A+B*X
A=-32.985299687
B= 43357.5000955

Source	Df	SS	MS	F
Regression	1	716104.698	716104.698	1539.677
Residual	3	1395.302	465.101	
Total	4	717500.000		

LINEAR



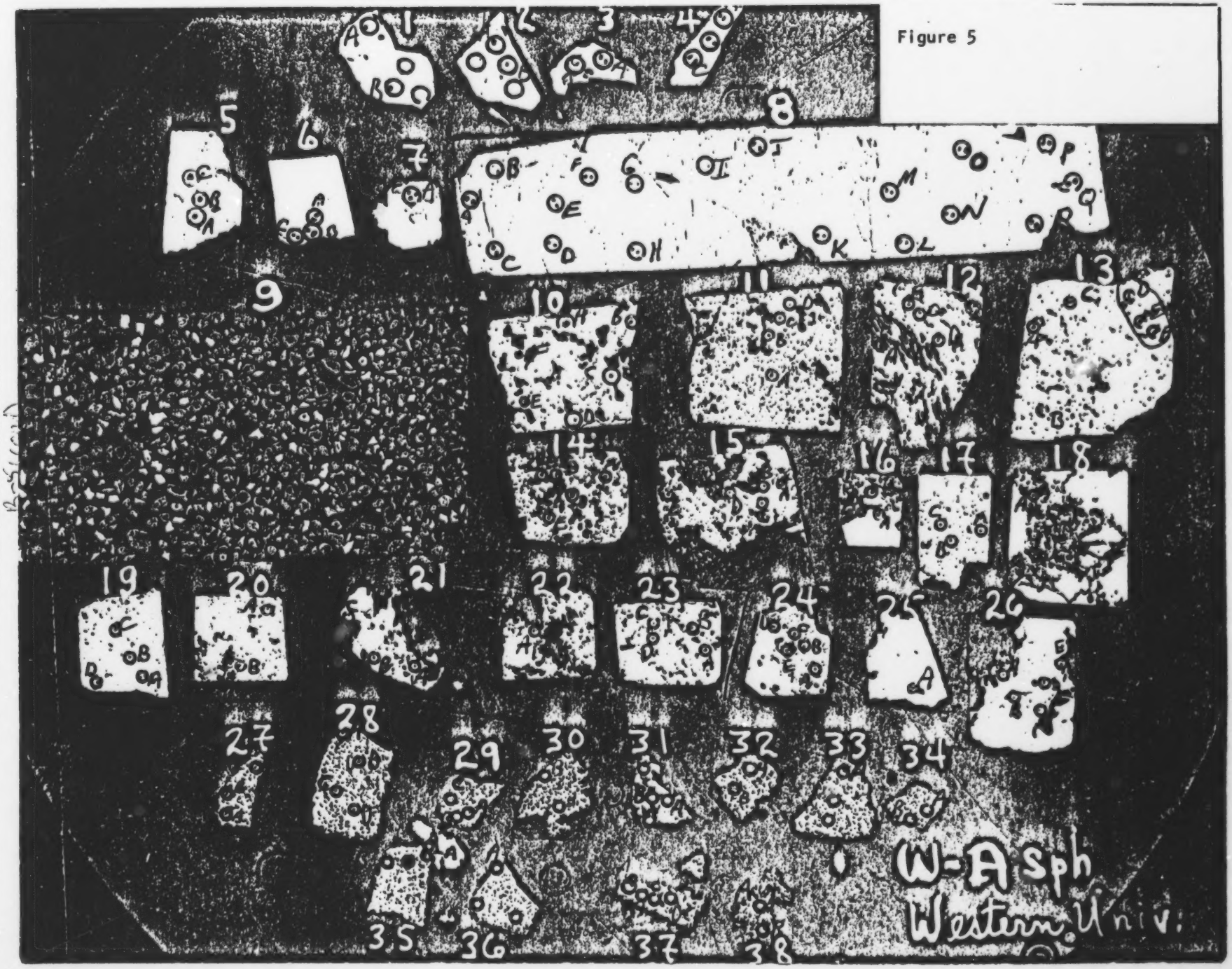


Figure 5

B-6

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

B-6

1984/01/24

Dr. Louis J. Cabri
Research & Technology
Canada Centre for Mineral
& Energy Technology
555 Booth Street
Ottawa, Ontario
K1A 0G1

4

Dear Louis,

I realized that I really hadn't given you a proper report of the December tests of your "A" and "B" polished specimens, sent on October 27th.

To date we have examined several of the synthetic sphalerite specimens from blocks polished by two different techniques. From the attached spectra we find:

(1) There is significantly more silver in straight ZnS-I(#10) than in straight ZnS-II(#6) or in ZnS containing 4.7% iron (#8).

(2) Polishing by the two different methods has no apparent effect on the blank silver content, but it does strongly affect the concentration of other trace elements detected. Polishing by method "A" leaves significantly higher concentrations of K, Na, Ca, Si, Fe, Cr, Mg and Zr.

We will check the PbS specimens shortly.

With best wishes.

Yours very truly,

N.S. McIntyre
Project Manager
Surface Science Western

B-7

File

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

B-7

1984/04/05

Dr. Louis J. Cabri
Canada Centre for Mineral &
~~Energy Technology~~
Canada Dept. of Energy Mines + Resources
~~555 Booth Street~~
Ottawa, Ont. M1A 0G1

Dear Louis,

I've enclosed some data which I hope will be useful for your purposes.

Firstly, a number of calibration plots are attached as Figures 1 - 7, including all those used in data analysis. The results of these are summarized below:

Series	Slope	Intercept	Data Analysis Application
III(5 points)	43357	-32.9	not used
IV (4 points)	37802	-5.5	Used for specimens 24, 25, 26, 27, 28, 29, 37 and 38 on 2/8/84. Also used for 30, 32 and 34 (in part) on 2/17/84 (see figure)
V (3 points)	34031	-0.7	Not used - poor reproducibility with Indium calibration on that day (2/17/84)
VI (3 points)	31648	3.4	Used for specimens 9 (in part), 10, 11, 12 and 13 on 2/24/84.
VII(3 points)	39364	-18.7	Used for 3, 4, 5, 6, 7, 9 (in part) on 3/5/84
VIII(3 points)	44998	-75.6	Used for 1, 2, 8, 13 on 3/12/84

Figure 7 is a plot showing calibration data taken on a number of different days, each one identified on the graph. It seems to me that the scatter for each point is random. Thus, there is probably a good case to be made for averaging all the calibration points to obtain a slope and intercept which could be considered valid for all the data. Let me know how you feel about this.

B-7

1984/04/05

Dr. Louis J. Cabri - 2 -

I've also enclosed some raw data for samples 1, 2, and 8, for you to use if you want to see the effect of using individual values for slope and intercept. Finally you will find the block with the line scan, which you requested, a new slide and xerox of the figure used to make the slide.

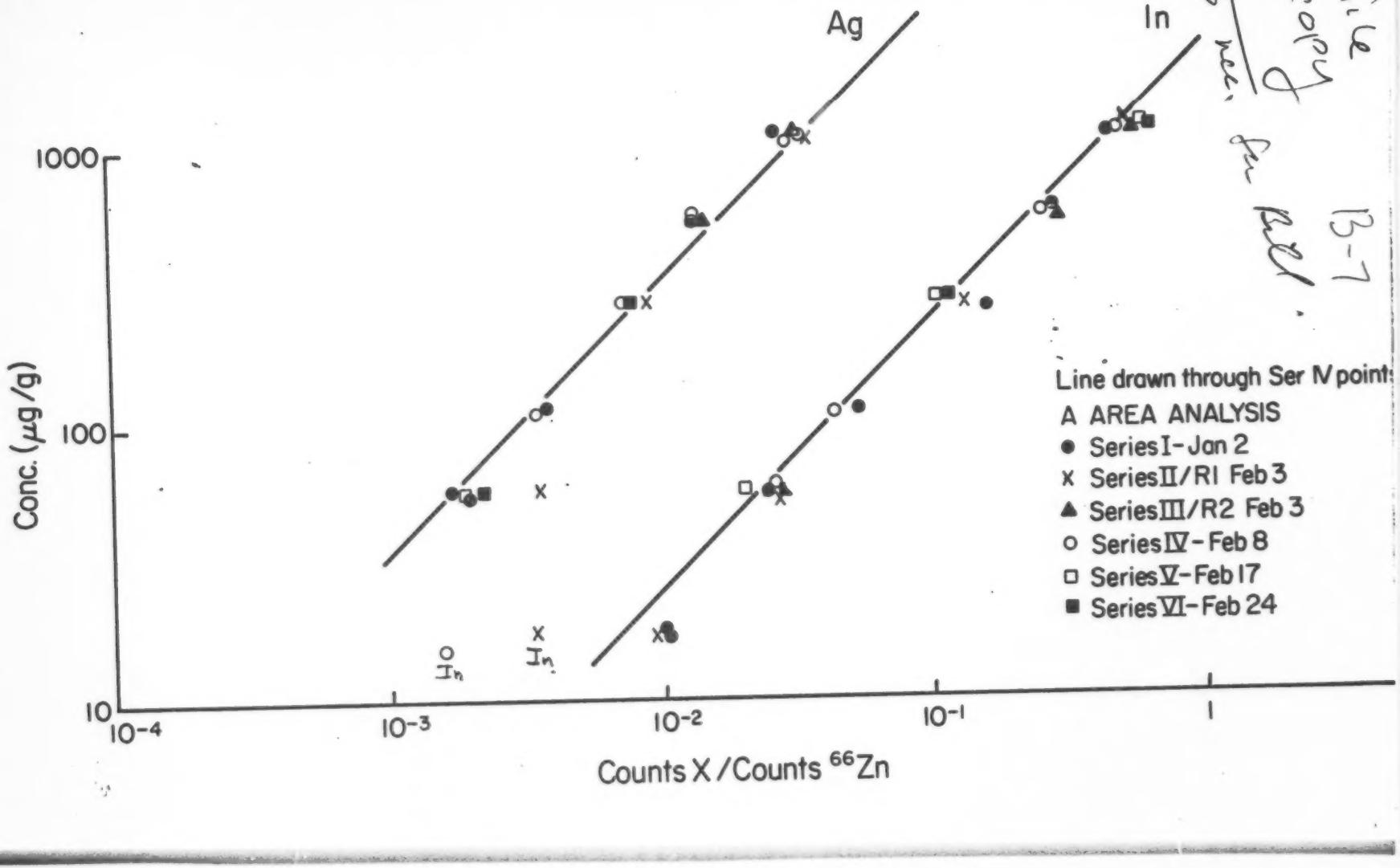
Best regards.

Yours very truly,

N.S. McIntyre
Director
Surface Science Western

cc: W.J. Chauvin

FILE: CDEMR



B For file copy
No copies req. for Bell.
B-7

B-7

DATA

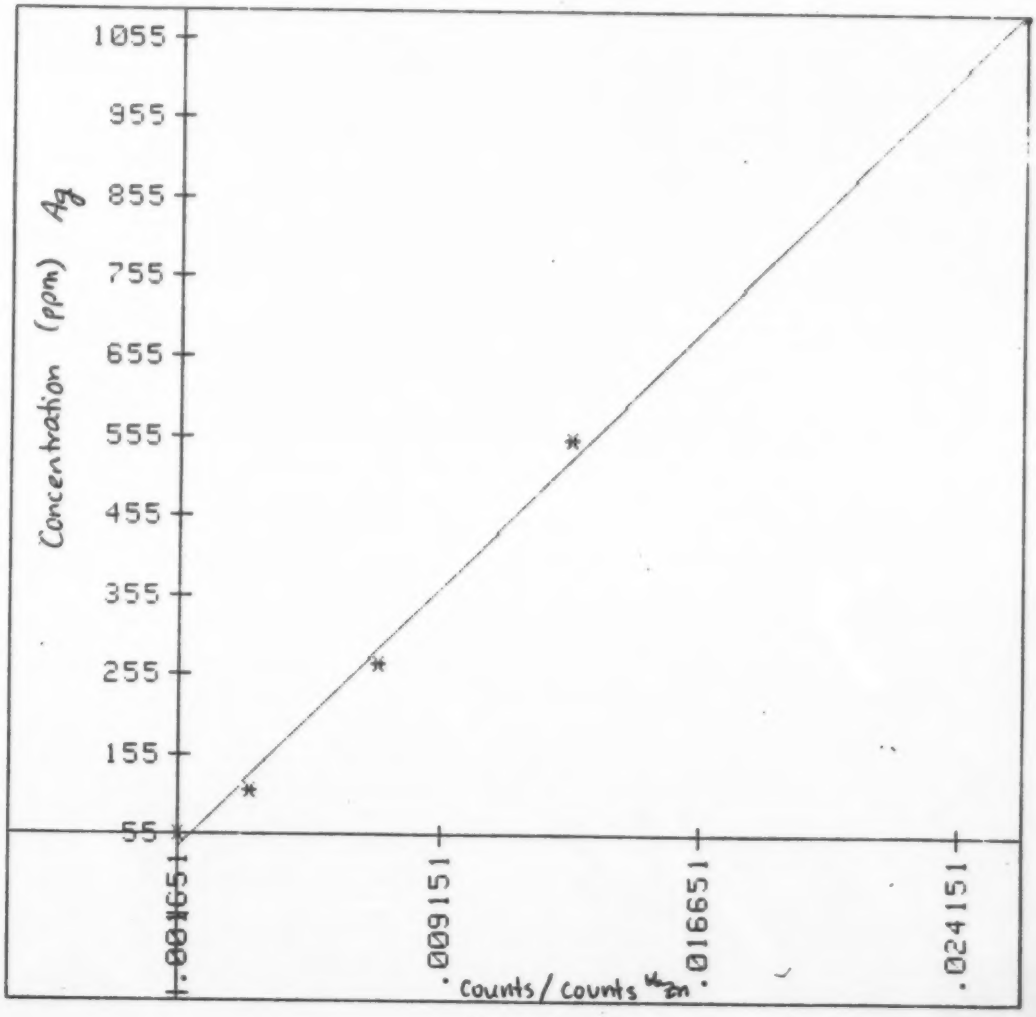
Point #1:	X=.001651	Y=55
Point #2:	X=.0036987	Y=110
Point #3:	X=.0073721	Y=270
Point #4:	X=.01292	Y=550
Point #5:	X=.02602	Y=1090

Ser III
-not used for any sample analysis

Linear model: Y=A+B*X
A=-32.985299687
B= 43357.5000955

Source	Df	SS	MS	F
Regression	1	716104.698	716104.698	1539.677
Residual	3	1395.302	465.101	
Total	4	717500.000		

LINEAR



s IV 8/2/84 B-7

Used for:
Specimens 24, 25, 26, 27, 28 29, 37 and 38 on Feb 8/84
Also adopted for:
Specimens, 30, 32, 34 (part) on Feb '17
This was used for Feb 17, data since the Endjum calibration curve on Feb. 17 showed poor reproducibility data

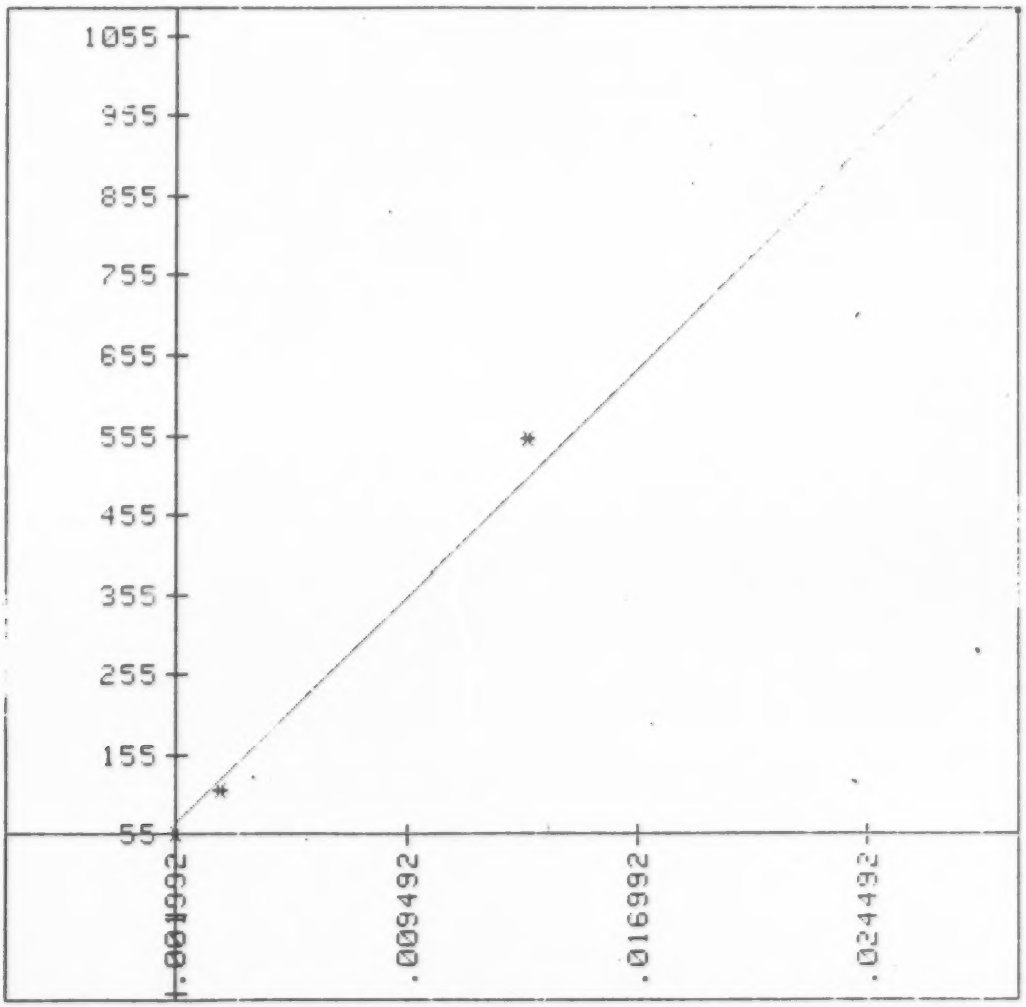
DATA

Point #1: 34	X=.001992	Y=55
Point #2: 33	X=.003429	Y=110
Point #3: 31	X=.01342	Y=550
Point #4: 30	X=.02949	Y=1090

Linear model: Y=A+B*X
A=-5.512135543
B= 37802.829285

S	Df	SS	MS	F
Regression	1	688105.480	688105.480	442.047
Residual	2	3113.270	1556.635	
Total	3	691218.750		

LINEAR



30, 32, 34-A-7

13-7

Point #1: X=.001964 Y=55
Point #2: X=.00746 Y=270
Point #3: X=.03209 Y=1090

Linear model: Y=A+B*X

A = .740903222

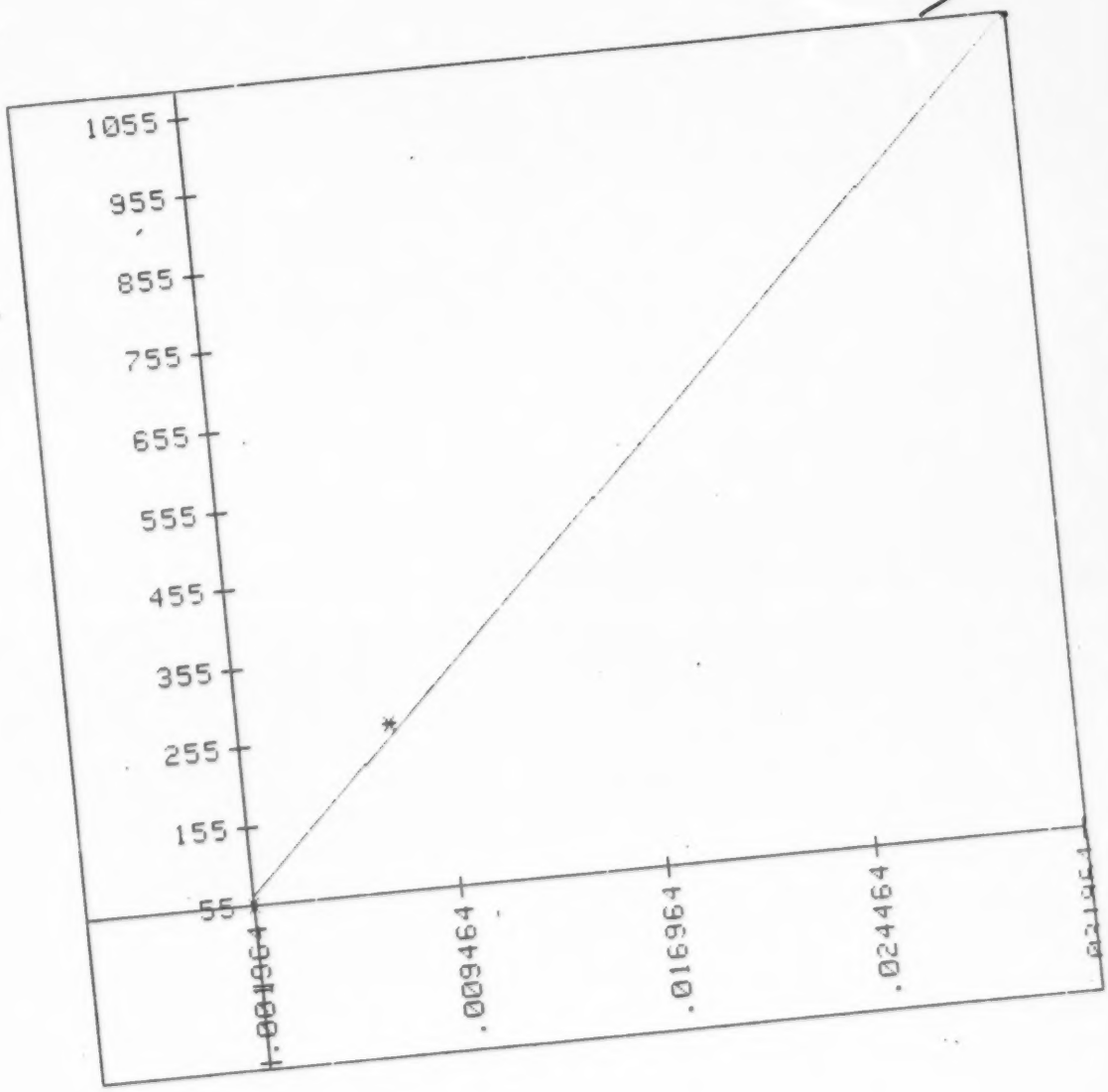
F = 34031.3459191

Source	Df	SS	MS	F
Regression	1	596213.865	596213.865	1480.166
Residual	1	402.802	402.802	
Total	2	596616.667		

~~Spe~~ This curve not used.

not used.

LINEAR



MASS:115 ATOMIC o/o

MASS: 66 PER CENT : +61.240
MASS:107 PER CENT : +1.965
PER CENT : +36.795

B-7 (cont'd)

24-FEB-84

~~30, 32, 34 1-8~~

Ag

9, 10, 11, 12, 13

part

Ser # VI

Used for 9, 10, 11, 12, 13

DATA

Point #1:	X=.002271	Y=55
Point #2:	X=.00765	Y=270
Point #3:	X=.03446	Y=1090

Linear model: Y=A+B*X

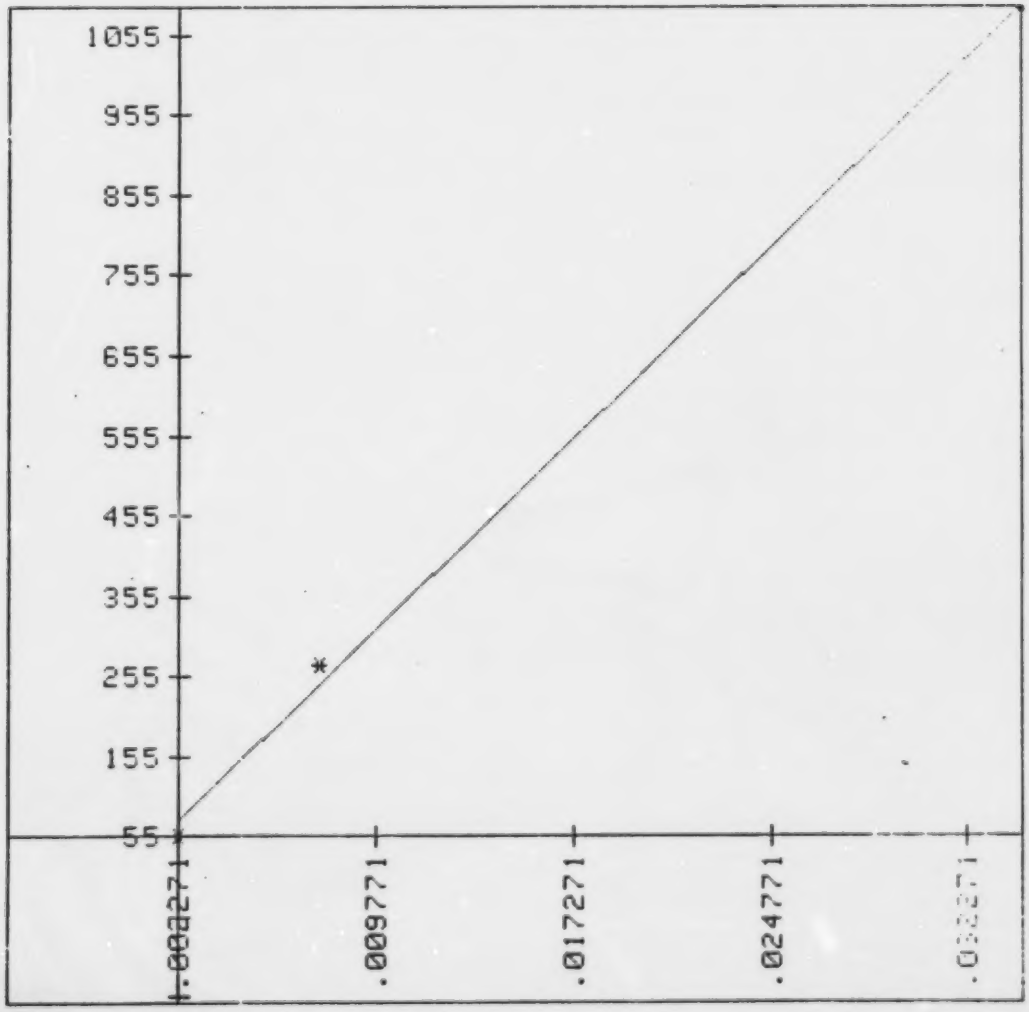
A= 3.466043892

B= 31648.7205859

Source	Df	SS	MS	F
Regression	1	595589.888	595589.888	580.057
Residual	1	1026.779	1026.779	
Total	2	596616.667		

Ag

LINEAR



ATOMIC o/o

MASS: 88 PER CENT : +97.944

B-7 (cond)
Mar 5/84 Ser VII
Used for
3, 4, 5, 6, 7, ~~8~~ 9,
part

DATA

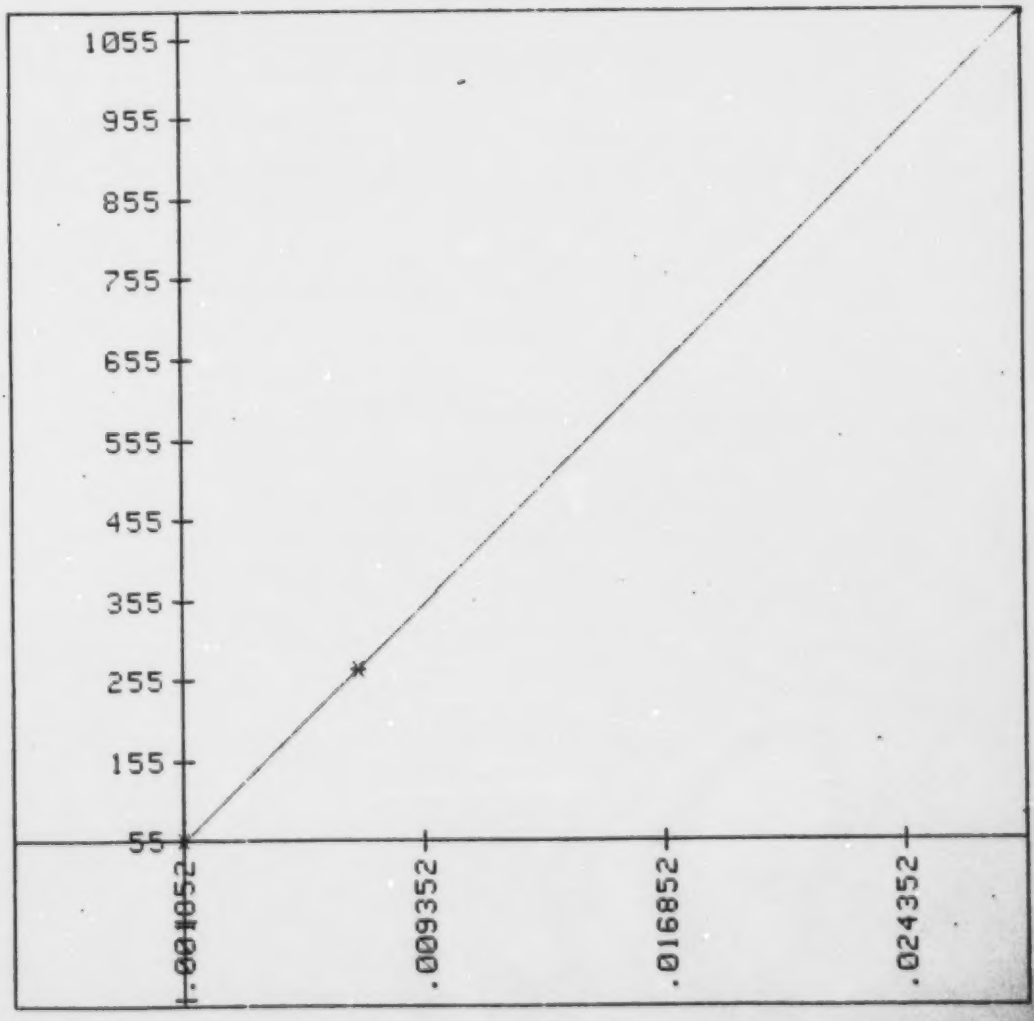
Point #1:	X=.001852	Y=55
Point #2:	X=.00736	Y=270
Point #3:	X=.02816	Y=1090

Linear model: Y=A+B*X
A=-18.704659384
B= 39364.0687723

Source	Df	SS	MS	F
Regression	1	596614.948	596614.948	347071.34486 M4D.3D
Residual	1	1.719	1.719	
Total	2	596616.667		

Ag

LINEAR



12/3/84

B-7 (cont'd)

Used for specimens 1, 2, 8, 13

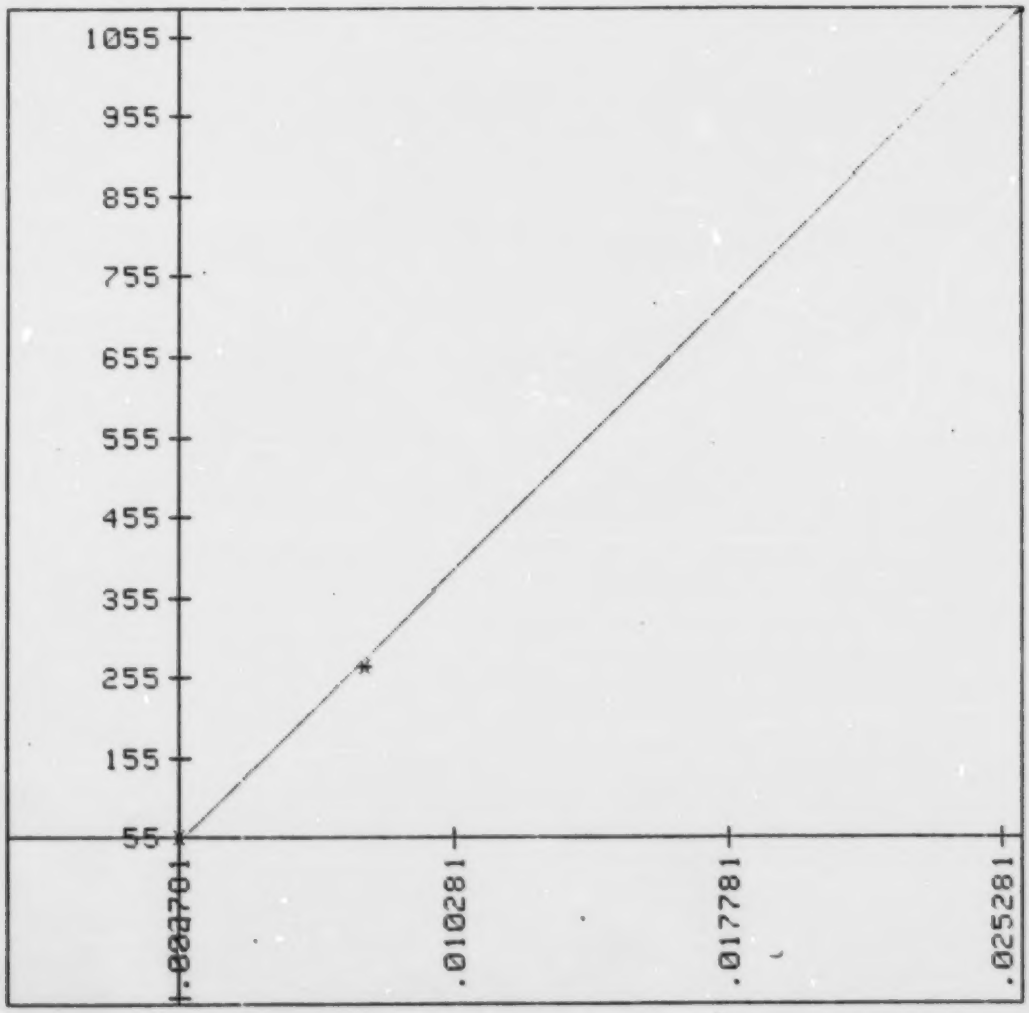
DATA

Point #1:	X=.002781	Y=55
Point #2:	X=.007839	Y=270
Point #3:	X=.02587	Y=1090

Linear model: Y=A+B*X
A=-75.66824866
B= 44998.7598242

Source	Df	SS	MS	F
Regression	1	596533.634	596533.634	7184.359
Residual	1	83.032	83.032	
Total	2	596616.667		

LINEAR



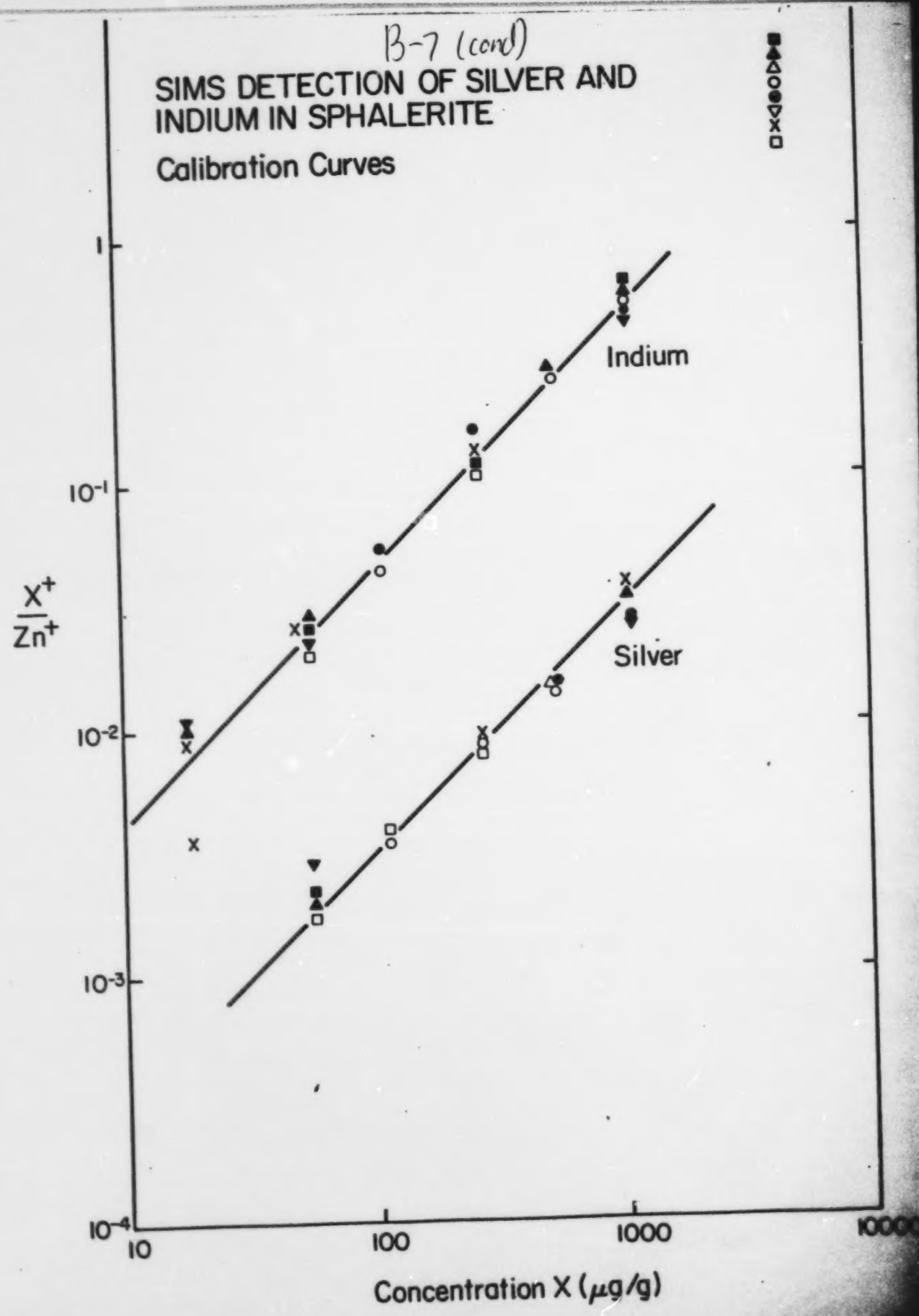
MASS:115 MEAN: +2.341E-02 STD DEV : +2.330E-04

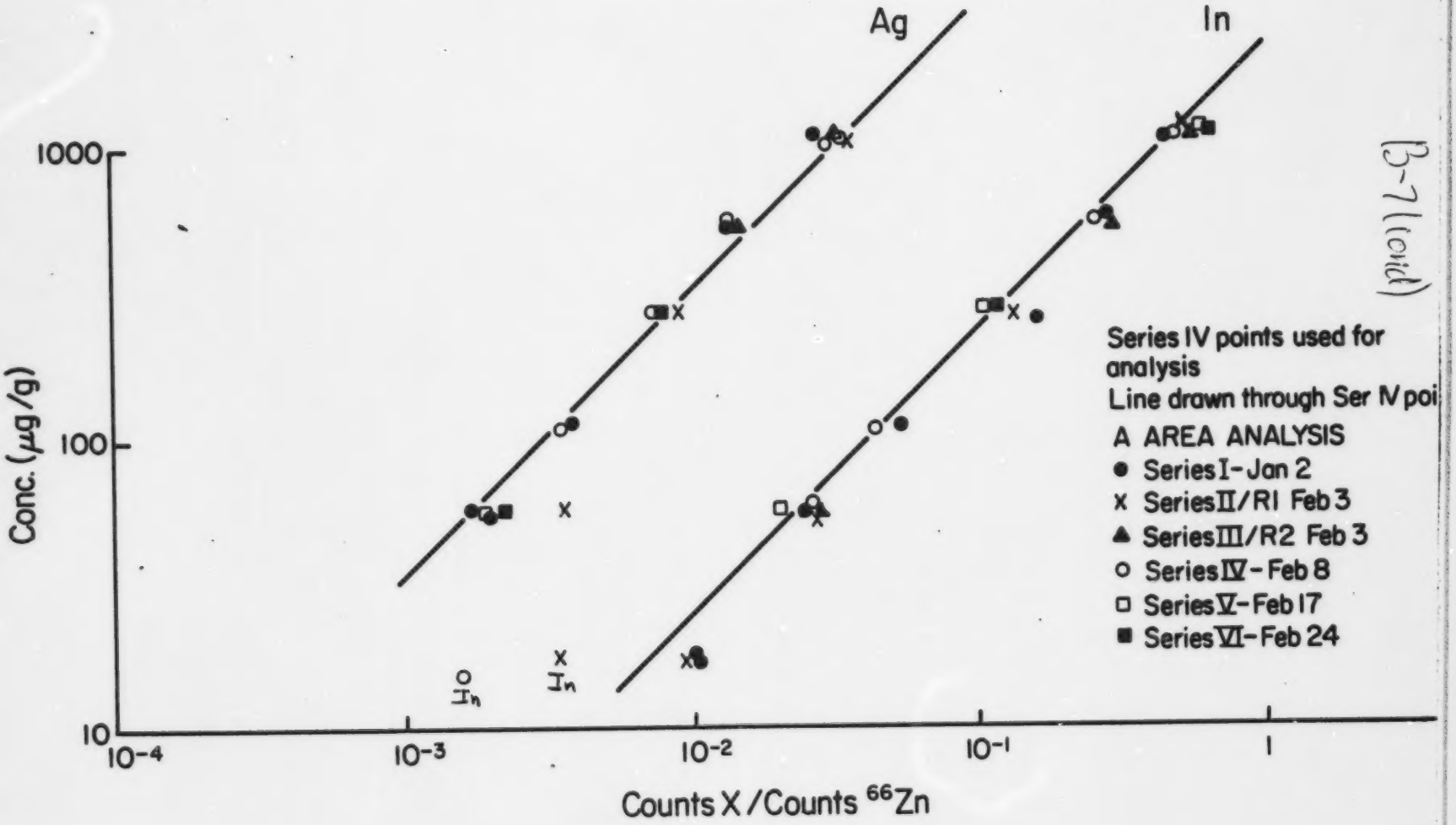
ATONIC o/o

MASS: 66 PER CENT : +97.448

MASS:107 PER CENT : +.271

B-7 (cont'd)


SIMS DETECTION OF SILVER AND INDIUM IN SPHALERITE
Calibration Curves
Indium
Silver
$\frac{X^+}{Zn^+}$
1
10^{-1}
10^{-2}
10^{-3}
10^{-4}
10
100
1000
10000
Concentration X (μg/g)


Ag
In
1000
100
10
Conc. (μg/g)
10-4
10-3
10-2
10-1
1
Counts X / Counts 66Zn
Series IV points used for analysis
Line drawn through Ser IV poi
A AREA ANALYSIS
Series I - Jan 2
Series II/R1 Feb 3
Series III/R2 Feb 3
Series IV - Feb 8
Series V - Feb 17
Series VI - Feb 24
In
In

B-7 (cont'd)

C

INVESTIGATIONS OF PRECISION AND ACCURACY IN SIMS ANALYSIS OF NATURAL SPHALERITES

(April 1984 - May 1985)

C-1

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

Research and Technology
Recherche et technologie

Canada Centre for Mineral and Energy Technology,
555 Booth Street
Ottawa, Ontario
K1A 0G1

Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa (Ontario)
K1A 0G1

Your file Votre référence

Our file Notre référence

SPECIAL DELIVERY

C-2

April 6, 1984

Dr. N.S. McIntyre,
Technical Director,
Surface Science,
Western Centre for Interdisciplinary
Studies in Chemical Physics,
University of Western Ontario
London, Ontario
N6A 3K7

Dear Stewart:

Further to my letter of April 5, I have been reviewing the analyses to date and would like you, please, to do the following analyses before you do the galenas.

Section A, dated 25/10/83 (see your letter 1984/01/24) ← B.G

Please analyse grains 6, 8, and 10 quantitatively in 5 different areas each. You will have to use the standards in Block W-A for your calibration curves and I don't know how changing "unknowns" and standards will affect your results. Please call if unclear or if any problems with this request.

Best wishes.

Yours sincerely,

Louis J. Cabri

LJC/tj

P.S. Just received your letter of 5 April - thanks. Also the polished section labeled 23/5/3 (L) which contains the pyrrhotite used for the ion-exchange experiments.

Canada

C-2

Energy, Mines and Resources Canada
Research and Technology
Canada Centre for Mineral and Energy Technology,
555 Booth Street
Ottawa, Ontario
K1A 0G1

Énergie, Mines et Ressources Canada
Recherche et technologie
Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa (Ontario)
K1A 0G1

Your file Votre référence

Our file Notre référence

C-2

April 9, 1984

Keep up 1, 2 & 8!

Dr. N.S. McIntyre,
Technical Director,
Surface Science,
Western Centre for Interdisciplinary
Studies in Chemical Physics,
University of Western Ontario,
London, Ontario
N6A 3K7

Dear Stewart:

I have now studied your report of March 1984 (letter of 23rd March) and the additional information with your letter of 5th April. I am, indeed, very disturbed by the negative gradient of Ag concentration for the majority of the unknowns. I have plotted several of these results for all 5 cycles (1-A-1, 1-B-1, 2-A-1, 2-B-1, 8-B-1, 8-B-2, 8-C-1, 8-C-2, 8-D-1, 8-D-2) and conclude that:

(a) The negative Ag concentration still has a steep gradient by the 5th cycle and the results are an uncertain quantity.
(b) There is no correlation of Ag concentration for two analyses in any particular circle.
(c) Some sort of Ag diffusion is produced under the ion beam which may be related to the grain size (in contrastto the standards) rather than heat of polishing as you suggest.

(d) While the precision of the results for the standards looks impressive, we cannot assign <u>any</u> concentrations for the unknowns.

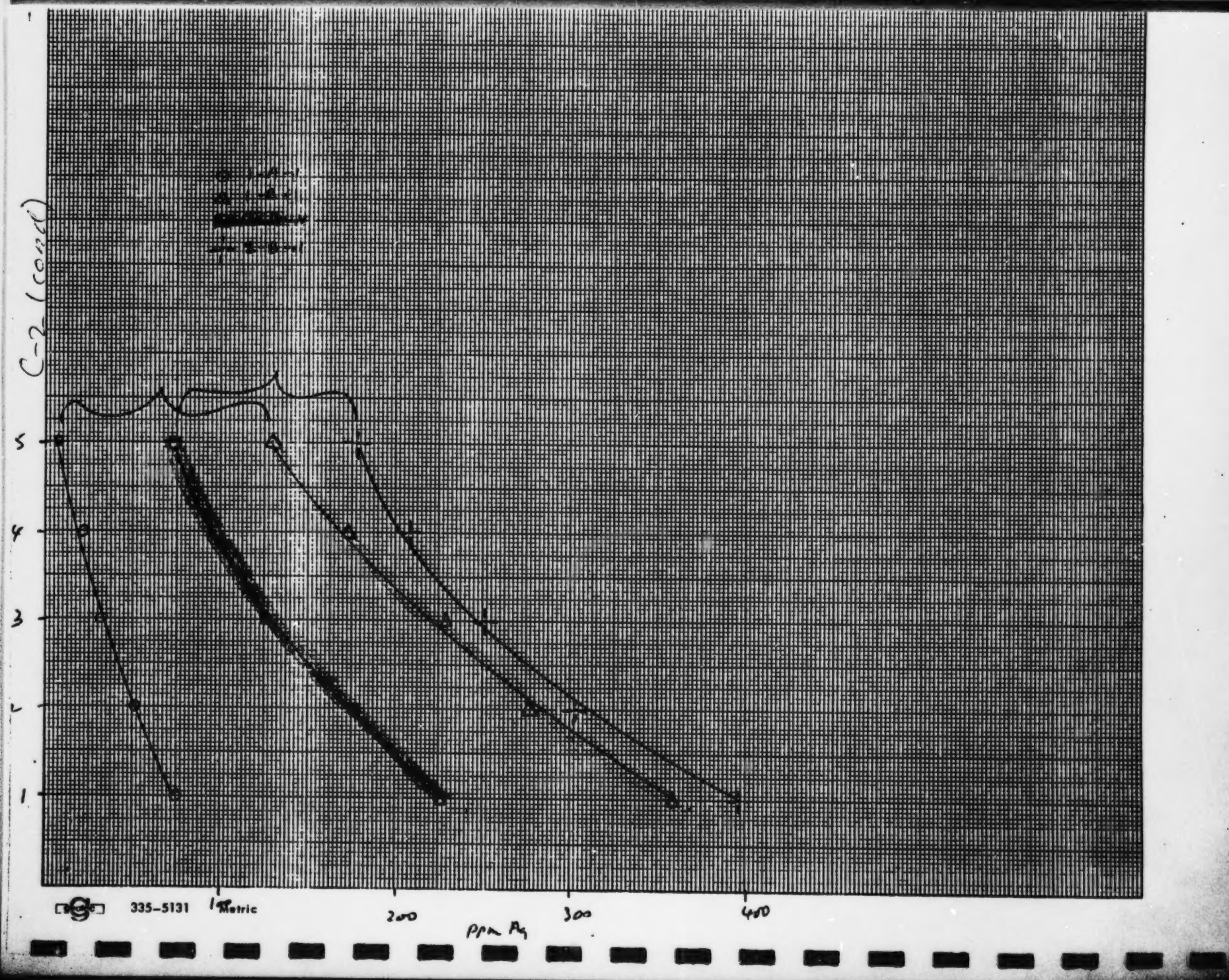
Obviously, a lot more work needs to be done in order to understand what is happening under the ion beam. I would like you please to return the sphalerite section (W-A sph) by return mail as I wish to re-study it very carefully. Best wishes.

Yours sincerely,

Louis J. Cabri

LJC/tj

Canada


C-2 (cont'd)
5
4
3
2
1
335-5131
Metric
200
300
400
ppm Ag

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

C-3

May 1, 1984

Dr. L. J. Cabri
Canada Centre for Mineral & Energy Technology
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis:

Contrary to what I told you earlier by phone, the earlier work we did on natural sphalerites did NOT exhibit a decrease in silver concentration from the outer surface into the bulk. The work I consulted earlier and reported to you turned out to be some early work on the WA block. The original data for the block submitted on October 27th is attached to this letter. It shows that, in contrast to the studies made on block WA, there is no significant change in silver/zinc ratio with depth.

Now the difference between these two series of experiments is the size (and density) of the primary beam. The original experiments were done with an O-beam of >30μm and a much higher beam density. Thus, I think that we should determine the difference in profiling with a large and a small beam on a large specimen such as WA-8. We shall also investigate the size and depth of the craters produced in the two series of experiments.

On May 4th therefore we shall be investigating block WA to determine:

(1) the effect of a long-term analysis with a small beam size, on a natural sphalerite.
(2) the effect of increased beam size & density on the silver/zinc ratio.
(3) Sulphur (mass 32) will also be monitered during the above analyses as you requested
(4) Crater depths will be measured and the resultant sputter rates will be used to relate derived silver concentrations to a specific depth into the natural sphalerite crystal.
(5) Effects of beam energy will also be studied.

I look forward to seeing you in a couple of weeks.

Yours very truly

N. S. Mc Intyre

C-3 (cont)

DATE	2/10/83
SAMPLE REFERENCE:	USP2A1
PRIMARY IONS:	OXYGEN
SECONDARY IONS:	
PRIMARY CURRENT:	59NANO AMP.
HIGH VOLTAGE:	11.9KILOVOLTS
RASTERING :	0 MICRONS
TRANSFER OPTICS :	150 MICRONS

MEASUREMENT CONDITIONS

MASS:	64
MASS:	107
MASS:	115
MASS REFERENCE:	64
	ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME:	5
WAITING TIME :	0
NUMBER OF CYCLES:	5
NUMBER OF BLOCKS:	1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->2.0664E+07
107/REF.-->	+3.868E-02	CNTS-->8.0514E+05
115/REF.-->	+2.857E-04	CNTS-->1.1951E+04
		BKGD-->6.050E+03

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->2.2402E+07
107/REF.-->	+4.075E-02	CNTS-->9.1829E+05
115/REF.-->	+2.504E-04	CNTS-->1.1260E+04
		BKGD-->5.652E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->2.3125E+07
107/REF.-->	+4.182E-02	CNTS-->9.7258E+05
115/REF.-->	+2.361E-04	CNTS-->1.1110E+04
		BKGD-->5.652E+03

CYCLE No: 4

64 REF.-->	+1.000E+00	CNTS-->2.2438E+
107 REF.-->	+4.313E-02	CNTS-->1.0160E+06
115 REF.-->	+2.372E-04	CNTS-->1.1064E+04
		BKGD-->5.505E+03

CYCLE No: 5

64 REF.-->	+1.000E+00	CNTS-->2.3705E+07
107/REF.-->	+4.395E-02	CNTS-->1.0470E+06
115/REF.-->	+2.220E-04	CNTS-->1.0580E+04
		BKGD-->5.319E+03

4.3 x 10^-2

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+4.167E-02
MASS:115	MEAN:	+2.463E-04

ATOMIC o/o

MASS: 64	PER CENT	:	+95.977
MASS:107	PER CENT	:	+3.999
MASS:115	PER CENT	:	+.024

HEIGHT (o o)

70 417

64
107
115

MASS: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

ZnS Results
Large O- beam
107 Ag
114 In

C-3 (recall

RESULTS

BLOCK No: 1

CYCLE No: 1

64 REF.-- +1.000E+00 CNTS--/6.843[illegible]E+06
[illegible] REF.-- +1.749E-02 CNTS--/2.1301E+05
115 REF. +3.802E-0[illegible] CNTS--/2.6603E+06
BKGD-- 9.496E+04

BLOCK No: 2

[illegible] +1.[illegible] CNTS--/6.8[illegible]E+06
[illegible] +1.[illegible] CNTS--/2.1415E+05
[illegible] [illegible] CNTS--/2.6[illegible]E+06
BKGD-- 9.[illegible]E+04

BLOCK No: 3

64 REF.-- [illegible]E+00 CNTS--/6.8068E+06
107 REF.-- +1.[illegible]E-02 CNTS-- 2.1778E+05
115 REF.-- +3.989E-01 CNTS--/2.7055E+06
BKGD--/9.268E+04

CYCLE No: 4

64 REF.-- +1.000E+00 CNTS-- 6.8451E+06
107 REF.-- [illegible]E-02 S-- 2.1998E+05
115 REF.-- +3.[illegible]E-01 [illegible]E+06
BKGD--/9.375E+04

CYCLE No: 5

64 REF.-- +1.000E+00 CNTS--/6.9712E+06
107 REF.-- +1.9[illegible]E-02 CNTS-- 2.1[illegible]E+05
115 REF.-- +3.[illegible]E-01 CNTS--[illegible]E+06
BKGD--/9.371E+04

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64 MEAN: +1.000E+00
MASS:107 MEAN: +1.849E-02
MASS:115 MEAN: +3.852E-01

ATOMIC o/o

MASS: 64 PER CENT : +71.241
MASS:107 PER CENT : +1.317
MASS:115 PER CENT : +27.442

WEIGHT (o/o)

MASS: 64 PER CENT : +58.036
MASS:107 PER CENT : +1.794
MASS:115 PER CENT : +40.170

MEASUREMENT CONDITIONS

DATE 10/4-83
SAMPLE REFERENCE: LC603AA

73mA
12kV

C-3 (cont'd)

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->6.0431E+06
107/REF.-->	+6.434E-03	CNTS-->7.1430E+04
115/REF.-->	+2.594E-01	CNTS-->1.5916E+06
		BKGD-->3.276E+04

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->7.4438E+06
107/REF.-->	+7.485E-03	CNTS-->8.8229E+04
115/REF.-->	+2.190E-01	CNTS-->1.6557E+06
		BKGD-->3.276E+04

#604
.05 SA.
457 ft

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->7.8458E+06
107/REF.-->	+8.433E-03	CNTS-->9.8641E+04
115/REF.-->	+2.192E-01	CNTS-->1.7453E+06
		BKGD-->3.276E+04

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->8.0237E+06
107/REF.-->	+8.771E-03	CNTS-->1.0285E+05
115/REF.-->	+2.202E-01	CNTS-->1.7935E+06
		BKGD-->3.276E+04

8.7

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->8.021[illegible]E+06
107/REF.-->	+9.00[illegible]E-03	CNTS-->1.0466E+05
115/REF.-->	+2.21[illegible]E-01	CNTS-->1.8051E+06
		BKGD-->3.276E+04

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64 MEAN: +1.000E+00
MASS:107 MEAN: +8.[illegible]5E-03
MASS:115 MEAN: +2.279E-01

ATOMIC o/o

MASS: 64 PER CENT : +80.908
MASS:107 PER CENT : +.649
MASS:115 PER CENT : +18.443

WEIGHT (o/o)

MASS: 64 PER CENT : +70.274
MASS:107 PER CENT : +.943
MASS:115 PER CENT : +28.784

MEASUREMENT CONDITIONS

DATE 10/4/83 MS AA
SAMPLE REFERENCE: LC604AA 12 AU
0-

C-3 (cont'd)

```
MASS:                64
MASS:               107
MASS:               115

MASS REFERENCE:      64
                ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME:     5
WAITING TIME :        0
NUMBER OF CYCLES:     5
NUMBER OF BLOCKS:     1
```

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->8.8216E+06
107/REF.-->	+1.591E-04	CNTS-->3.1480E+03
115/REF.-->	+4.191E-04	CNTS-->5.4410E+03
		BKGD-->1.745E+03

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->9.5211E+06
107/REF.-->	+1.147E-04	CNTS-->2.3230E+03
115/REF.-->	+4.446E-04	CNTS-->5.4640E+03
		BKGD-->1.231E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->9.5116E+06
107/REF.-->	+9.579E-05	CNTS-->1.8760E+03
115/REF.-->	+4.702E-04	CNTS-->5.4370E+03
		BKGD-->9.650E+02

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->9.4369E+06
107/REF.-->	+8.616E-05	CNTS-->1.6570E+03
115/REF.-->	+4.882E-04	CNTS-->5.4510E+03
		BKGD-->8.440E+02

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->9.4441E+06
107/REF.-->	+8.059E-05	CNTS-->1.5930E+03
115/REF.-->	+4.916E-04	CNTS-->5.4740E+03
		BKGD-->8.320E+02

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+1.073E-04
MASS:115	MEAN:	+4.627E-04

ATOMIC o/o

MASS: 64	PER CENT	:	+99.943
MASS:107	PER CENT	:	+.011
MASS:115	PER CENT	:	+.046

WEIGHT (o/o)

MASS: 64	PER CENT	:	+99.899
MASS:107	PER CENT	:	+.018
MASS:115	PER CENT	:	+.083

MEASUREMENT CONDITIONS

DATE 10/4/83 12 KV

SAMPLE REFERENCE: LC605AA

C-3 (cont)

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.--> +1.000E+00 CNTS-->6.5815E+06
107/REF.--> +3.425E-03 CNTS-->4.2125E+04
115/REF.--> +1.365E-01 CNTS-->9.1506E+05
BKGD-->1.965E+04

CYCLE No: 2

64/REF.--> +1.000E+00 CNTS-->7.9808E+06
107/REF.--> +3.877E-03 CNTS-->5.0516E+04
115/REF.--> +1.155E-01 CNTS-->9.3885E+05
BKGD-->1.965E+04

CYCLE No: 3

64/REF.--> +1.000E+00 CNTS-->8.2194E+06
107/REF.--> +4.405E-03 CNTS-->5.5771E+04
115/REF.--> +1.167E-01 CNTS-->9.7617E+05
BKGD-->1.965E+04

CYCLE No: 4

64/REF.--> +1.000E+00 CNTS-->8.2937E+06
107/REF.--> +4.889E-03 CNTS-->6.0096E+04
115/REF.--> +1.219E-01 CNTS-->1.0282E+06
BKGD-->1.965E+04

CYCLE No: 5

64/REF.--> +1.000E+00 CNTS-->8.4510E+06
107/REF.--> +5.065E-03 CNTS-->6.2516E+04
115/REF.--> +1.248E-01 CNTS-->1.0722E+06
BKGD-->1.9[illegible]E+04

Rh.

REFERENCE PEAK MASS: 64

MASS: 64 MEAN: +1.000E+00
MASS:107 MEAN: [illegible]
MASS:115 MEAN: +1.231E-01

ATOMIC o/o

MASS: 64 PER CENT : +88.700
MASS:107 PER CENT : +.385
MASS:115 PER CENT : +10.915

WEIGHT (o/o)

MASS: 64 PER CENT : +81.489
MASS:107 PER CENT : +.590
MASS:115 PER CENT : +18.001

MEASUREMENT CONDITIONS

DATE 10-4-83

C-3 (land)

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.0661E+07
107/REF.-->	+1.367E-03	CNTS-->2.7900E+04
115/REF.-->	+4.017E-02	CNTS-->4.4100E+05
		BKGD-->1.335E+04

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.2150E+07
107/REF.-->	+1.549E-03	CNTS-->3.2152E+04
115/REF.-->	+4.091E-02	CNTS-->5.0982E+05
		BKGD-->1.335E+04

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.2903E+07
107/REF.-->	+1.742E-03	CNTS-->3.5799E+04
115/REF.-->	+4.169E-02	CNTS-->5.5074E+05
		BKGD-->1.335E+04

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.3498E+07
107/REF.-->	+1.835E-03	CNTS-->3.8092E+04
115/REF.-->	+4.406E-02	CNTS-->6.0745E+05
		BKGD-->1.335E+04

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.3447E+07
107/REF.-->	+1.766E-03	CNTS-->3.7073E+04
115/REF.-->	+4.288E-02	CNTS-->5.8937E+05
		BKGD-->1.335E+04

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+1.692E-03
MASS:115	MEAN:	+4.194E-02

ATOMIC %

MASS: 64	PER CENT	:	+95.823
MASS:107	PER CENT	:	+.158
MASS:115	PER CENT	:	+4.019

WEIGHT (%)

MASS: 64	PER CENT	:	+92.754
MASS:107	PER CENT	:	+.256
MASS:115	PER CENT	:	+6.990

MEASUREMENT CONDITIONS

DATE 10/4/83
SAMPLE REFERENCE: LC607AA
PRINT [illegible]: 0

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

Zinc Sphalerite Unknowns.

C-3 (cont'd)

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.9095E+07
107/REF.-->	+6.838E-04	CNTS-->1.9005E+04
115/REF.-->	+3.088E-04	CNTS-->1.1862E+04
		BKGD-->5.968E+03

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->2.0807E+07
107/REF.-->	+6.726E-04	CNTS-->1.9457E+04
115/REF.-->	+2.773E-04	CNTS-->1.1233E+04
		BKGD-->5.465E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->2.1243E+07
107/REF.-->	+7.251E-04	CNTS-->2.0864E+04
115/REF.-->	+2.604E-04	CNTS-->1.0996E+04
		BKGD-->5.465E+03

CYCLE No: 4

64/REF	+1.000E+00	CNTS-->2.1352E+07
[illegible]	[illegible]	CNTS-->2.5321E+04
[illegible]	[illegible]	CNTS-->1.0674E+04
		BKGD-->5.248E+03

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->2.1470E+07
107/REF.-->	+9.302E-04	CNTS-->2.5166E+04
115/REF.-->	+2.408E-04	CNTS-->1.0540E+04
		BKGD-->5.200E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+7.902E-04
MASS:115	MEAN:	+2.699E-04

ATOMIC % %

MASS: 64	PER CENT :	+99.894
MASS:107	PER CENT :	+.079
MASS:115	PER CENT :	-.027

WEIGHT (%/%)

MASS: 64	PER CENT :	+99.828
MASS:107	PER CENT :	+.132
MASS:115	PER CENT :	+.048

MEASUREMENT CONDITION:

DAT
SAM REFERENCE: USPSA1

C-3 (cont'd)

```
MASS:          64
MASS:         107
MASS:         115

MASS REFERENCE:    64
              ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME:   5
WAITING TIME :      0
NUMBER OF CYCLES:   5
NUMBER OF BLOCKS:   1
```

RESULTS

BLOCK No: 1

CYCLE No: 1

```
64/REF.-->    +1.000E+00        CNTS-->1.8024E+07
107/REF.-->   +1.043E-03 <      CNTS-->2.4981E+04
115/REF.-->   +3.305E-04        CNTS-->1.2041E+04
                                BKGD-->6.186E+03
```

USP 4A1

CYCLE No: 2

```
64/REF.-->    +1.000E+00        CNTS-->1.9562E+07
107/REF.-->   +1.043E-03 <      CNTS-->2.5038E+04
115/REF.-->   +2.878E-04        CNTS-->1.1075E+04
                                BKGD-->5.447E+03
```

CYCLE No: 3

```
64/REF.-->    +1.000E+00        CNTS-->2.0057E+07
107/REF.-->   +1.078E-03 <      CNTS-->2.6902E+04
115/REF.-->   +2.723E-04        CNTS-->1.0908E+04
                                BKGD-->5.447E+03
```

CYCLE No: 4

```
64/REF.-->    +1.000E+00        CNTS-->2.0228E+07
107/REF.-->   +1.081E-03 <      CNTS-->2.7260E+04
115/REF.-->   +2.604E-04        CNTS-->1.0671E+04
                                BKGD-->5.405E+03
```

CYCLE No: 5

```
64/REF.-->    +1.000E+00        CNTS-->2.0183E+07
107/REF.-->   +1.147E-03 <      CNTS-->2.7857E+04
115/REF.-->   +2.451E-04        CNTS-->9.7010E+03
                                BKGD-->4.755E+03
```

1.1 x 10^-3

MEAN RATIO

```
REFERENCE PEAK MASS:  64

MASS: 64      MEAN:    +1.000E+00
MASS:107      MEAN:    +1.077E-03
MASS:115      MEAN:    +2.792E-04
```

ATOMIC o/o

```
MASS: 64    PER CENT  :  +99.865
MASS:107    PER CENT  :    +.108
MASS:115    PER CENT  :    +.028
```

WEIGHT (o/o)

```
MASS: 64    PER CENT  : +99.770
MASS:107    PER CENT  :   +.180
MASS:115    PER CENT  :   +.050
```

MEASUREMENT CONDITIONS

```
DATE                    2/10/83
SAMPLE REFERENCE:       USP4A1
```

67 hr 0
12 hr

C-3 (cont'd)

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.7084E+07
107/REF.-->	+7.561E-04 ←	CNTS-->1.6052E+04
115/REF.-->	+2.966E-04	CNTS-->1.0204E+04
		BKGD-->5.138E+03

USIP 2A!

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.8006E+07
107/REF.-->	+7.184E-04 ←	CNTS-->1.7594E+04
115/REF.-->	+2.650E-04	CNTS-->9.4318E+03
		BKGD-->4.661E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.8346E+07
107/REF.-->	+7.296E-04 ←	CNTS-->1.7811E+04
115/REF.-->	+2.413E-04	CNTS-->8.8540E+03
		BKGD-->4.429E+03

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.8450E+07
107/REF.-->	+7.213E-04 ←	CNTS-->1.7601E+04
115/REF.-->	+2.349E-04	CNTS-->8.6290E+03
		BKGD-->4.296E+03

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.8535E+07
107/REF.-->	+7.601E-04 ←	CNTS-->1.8181E+04
115/REF.-->	+2.278E-04	CNTS-->8.3170E+03
		BKGD-->4.096E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64 MEAN: +1.000E+00
MASS:107 MEAN: +7.371E-04 7.4×10⁻⁴
MASS:115 MEAN: +2.531E-04

ATOMIC o/o

MASS: 64 PER CENT : +99.901
MASS:107 PER CENT : +.074
MASS:115 PER CENT : +.025

WEIGHT (o/o)

MASS: 64 PER CENT : +99.832
MASS:107 PER CENT : +.123
MASS:115 PER CENT : +.045

MEASUREMENT CONDITIONS

DATE 2/10/83 5 PM 0-

C-3(cond)

```
MASS:          64
MASS:         107
MASS:         115

MASS REFERENCE:    64
              ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME:   5
WAITING TIME :      0
NUMBER OF CYCLES:   5
NUMBER OF BLOCKS:   1
```

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.7452E+07
107/REF.-->	+1.335E-03	CNTS-->2.7757E+04
115/REF.-->	+2.601E-04	CNTS-->8.9990E+03
		BKGD-->4.460E+03

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.8546E+07
107/REF.-->	+1.392E-03	CNTS-->3.0899E+04
115/REF.-->	+2.302E-04	CNTS-->8.5660E+03
		BKGD-->4.297E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.8795E+07
107/REF.-->	+1.442E-03	CNTS-->3.1378E+04
115/REF.-->	+2.312E-04	CNTS-->8.6270E+03
		BKGD-->4.282E+03

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.8922E+07
107/REF.-->	+1.513E-03	CNTS-->3.2668E+04
115/REF.-->	+2.269E-04	CNTS-->8.3430E+03
		BKGD-->4.051E+03

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.8922E+07
107/REF.-->	+1.596E-03	CNTS-->3.4192E+04
115/REF.-->	+2.144E-04	CNTS-->8.0520E+03
		BKGD-->3.996E+03

MEAN RATIO

```
REFERENCE PEAK MASS:   64

MASS: 64       MEAN:       +1.000E+00
MASS:107       MEAN:       +1.456E-03
MASS:115       MEAN:       +2.326E-04
```

ATOMIC o/o

```
MASS: 64     PER CENT   :  +99.831
MASS:107     PER CENT   :    +.145
MASS:115     PER CENT   :    +.023
```

WEIGHT (o/o)

```
MASS: 64     PER CENT   : +99.716
MASS:107     PER CENT   :   +.243
MASS:115     PER CENT   :   +.042
```

MEASUREMENT CONDITIONS

```
DATE                  2/10/83
SAMPLE REFERENCE:     USP3A1
PRIMARY IONS:         OXYGEN
```

C-3 (cont'd)

```
MASS:                64
MASS:               107
MASS:               115

MASS REFERENCE:      64
                ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME:     5
WAITING TIME :        0
NUMBER OF CYCLES:     5
NUMBER OF BLOCKS:     1
```

```
                        RESULTS
```

```
                        BLOCK No:    1

                      CYCLE No:  1

     64/REF.-->      +1.000E+00          CNTS-->2.0664E+07
    107/REF.-->      +3.869E-02          CNTS-->8.0514E+05
    115/REF.-->      +2.857E-04          CNTS-->1.1951E+04
                                         BKGD-->6.050E+03

                      CYCLE No:  2

     64/REF.-->      +1.000E+00          CNTS-->2.2402E+07
    107/REF.-->      +4.075E-02          CNTS-->9.1829E+05
    115/REF.-->      +2.504E-04          CNTS-->1.1260E+04
                                         BKGD-->5.652E+03

                      CYCLE No:  3

     64/REF.-->      +1.000E+00          CNTS-->2.3125E+07
    107/REF.-->      +4.182E-02          CNTS-->9.7258E+05
    115/REF.-->      +2.361E-04          CNTS-->1.1110E+04
                                         BKGD-->5.652E+03

                      CYCLE No:  4

     64/REF.-->      +1.000E+00          CNTS-->2.3438E+07
    107/REF.-->      +4.313E-02          CNTS-->1.0160E+06
    115/REF.-->      +2.372E-04          CNTS-->1.1064E+04
                                         BKGD-->5.505E+03

                      CYCLE No:  5

     64/REF.-->      +1.000E+00          CNTS-->2.3705E+07
    107/REF.-->      +4.395E-02          CNTS-->1.0470E+06
    115/REF.-->      +2.220E-04          CNTS-->1.0580E+04
                                         BKGD-->5.319E+03
```

4.3 x 10^-2

```
                      MEAN RATIO

     REFERENCE PEAK MASS:   64

     MASS: 64       MEAN:        +1.000E+00
     MASS:107       MEAN:        +4.167E-02
     MASS:115       MEAN:        +2.463E-04

                      ATOMIC  o/o

     MASS: 64    PER CENT   :  +95.977
     MASS:107    PER CENT   :    +3.999
     MASS:115    PER CENT   :      +.024

                      HEIGHT (o/o)

     MASS: 64    PER CENT   : +93.449
     MASS:107    PER CENT   :  +6.510
     MASS:115    PER CENT   :    +.041
```

```
          MEASUREMENT CONDITIONS
     DATE                2/10/83
     SAMPLE REFERENCE:   USP2A2
     REMARK LINE:        OXYGEN
```

C-31 (cont'd)

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.7884E+07
107/REF.-->	+7.561E-04	CNTS-->1.8852E+04
115/REF.-->	+2.966E-04	CNTS-->1.0284E+04
		BKGD-->5.138E+03

USP 2A1

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.8006E+07
107/REF.-->	+7.184E-04	CNTS-->1.7594E+04
115/REF.-->	+2.650E-04	CNTS-->9.4310E+03
		BKGD-->4.661E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.8346E+07
107/REF.-->	+7.296E-04	CNTS-->1.7811E+04
115/REF.-->	+2.413E-04	CNTS-->8.8540E+03
		BKGD-->4.429E+03

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.8450E+07
107/REF.-->	+7.213E-04	CNTS-->1.7681E+04
115/REF.-->	+2.349E-04	CNTS-->8.6290E+03
		BKGD-->4.296E+03

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.8535E+07
107/REF.-->	+7.601E-04	CNTS-->1.8181E+04
115/REF.-->	+2.278E-04	CNTS-->8.3170E+03
		BKGD-->4.096E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+7.371E-04
MASS:115	MEAN:	+2.531E-04

7.4 x 10⁻⁴

ATOMIC o/o

MASS: 64	PER CENT :	+99.981
MASS:107	PER CENT :	+.074
MASS:115	PER CENT :	+.025

WEIGHT (o/o)

MASS: 64	PER CENT :	+99.832
MASS:107	PER CENT :	+.123
MASS:115	PER CENT :	+.045

MEASUREMENT CONDITIONS

DATE 2/10/83
SAMPLE REFERENCE: USP2A1

54mA 12kV

C-3 (cont'd)

DATE 2/10/83
SAMPLE REFERENCE: USP2A2

PRIMARY IONS: OXYGEN -
SECONDARY IONS: +

PRIMARY CURRENT: 70NANO AMP.
HIGH VOLTAGE: 11.9KILOVOLTS
RASTERING : 0 MICRONS
TRANSFER OPTICS : 150 MICRONS

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.6402E+07
107/REF.-->	+5.403E-04	CNTS-->1.4128E+04
115/REF.-->	+3.127E-04	CNTS-->1.0245E+04
		BKGD-->5.093E+03

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.7493E+07
107/REF.-->	+4.816E-04	CNTS-->1.3285E+04
115/REF.	+2.851E-04	CNTS-->9.8490E+03
		BKGD-->4.863E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.7868E+07
107/REF.-->	+4.000E-04	CNTS-->1.3153E+04
115/REF.-->	+2.710E-04	CNTS-->9.4330E+03
		BKGD-->4.570E+03

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.7977E+07
107/REF.-->	+4.769E-04	CNTS-->1.3007E+04
115/REF.-->	+2.517E-04	CNTS-->8.9600E+03
		BKGD-->4.436E+03

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.8016E+07
107/REF.-->	+5.012E-04	CNTS-->1.3282E+04
115/REF.-->	+2.547E-04	CNTS-->8.8430E+03
		BKGD-->4.255E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+4.976E-04
MASS:115	MEAN:	+2.752E-04

ATOMIC o/o

MASS: 64	PER CENT	:	+99.923
MASS:107	PER CENT	:	+.050
MASS:115	PER CENT	:	+.027

WEIGHT (o/o)

MASS: 64	PER CENT	:	+99.868
MASS:107	PER CENT	:	+.093

C-3 (cond)

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.7323E+07
107/REF.-->	+2.070E-03	CNTS-->4.1001E+04
115/REF.-->	+2.985E-04	CNTS-->1.0315E+04
		BKGD-->5.146E+03

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.8715E+07
107/REF.-->	+2.143E-03	CNTS-->4.4912E+04
115/REF.-->	+2.591E-04	CNTS-->9.6200E+03
		BKGD-->4.773E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.9217E+07
107/REF.-->	+2.201E-03	CNTS-->4.6850E+04
115/REF.-->	+2.453E-04	CNTS-->9.2760E+03
		BKGD-->4.563E+03

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.9362E+07
107/REF.-->	+2.298E-03	CNTS-->4.8819E+04
115/REF.-->	+2.246E-04	CNTS-->8.8360E+03
		BKGD-->4.408E+03

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.9525E+07
107/REF.-->	+2.350E-03	CNTS-->5.0171E+04
115/REF.-->	+2.461E-04	CNTS-->9.0940E+03
		BKGD-->4.289E+03

2.3x.3

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+2.211E-03
MASS:115	MEAN:	+2.547E-04

ATOMIC o/o

MASS: 64	PER CENT :	+99.754
MASS:107	PER CENT :	+.221
MASS:115	PER CENT :	+.025

WEIGHT (o/o)

MASS: 64	PER CENT :	+99.586
MASS:107	PER CENT :	+.368
MASS:115	PER CENT :	+.046

MEASUREMENT CONDITIONS

C-3A

C-3 (cont'd)

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

Calibration

RESULTS

BLOCK No: 1

Calib Point 1

CYCLE No: 1

64/REF.--> +1.000E+00 CNTS-->8.3562E+06
107/REF.--> +1.405E-02 CNTS-->2.2709E+05
115/REF.--> +5.994E-01 CNTS-->5.0532E+06
BKGD-->1.113E+05

CYCLE No: 2

64/REF.--> +1.000E+00 CNTS-->8.5205E+06
107/REF.--> +1.438E-02 CNTS-->2.3222E+05
115/REF.--> +6.008E-01 CNTS-->5.1633E+06
BKGD-->1.113E+05

CYCLE No: 3

64/REF.--> +1.000E+00 CNTS-->8.5196E+06
107/REF.--> +1.544E-02 CNTS-->2.4107E+05
115/REF.--> +6.172E-01 CNTS-->5.3006E+06
BKGD-->1.113E+05

CYCLE No: 4

64/REF.--> +1.000E+00 CNTS-->8.509[illegible]
107/REF.--> +1.538E-02 CNTS-->2.404[illegible]
115/REF.--> +6.388E-01 CNTS-->5.4089E+06
BKGD-->1.113E+05

CYCLE No: 5

64/REF.--> +1.000E+00 CNTS-->8.4559E+06
107/REF.--> +1.602E-02 CNTS-->2.4491E+05
115/REF.--> +6.454E-01 CNTS-->5.4968E+06
BKGD-->1.113E+05

0.1% Ag
0.1% In

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64 MEAN: +1.000E+00
MASS:107 MEAN: +1.505E-02
MASS:115 MEAN: +6.187E-01

ATOMIC o/o

MASS: 64 PER CENT : +61.209
MASS:107 PER CENT : +.921
MASS:115 PER CENT : +37.870

WEIGHT (o/o)

MASS: 6[illegible] PER CENT : +46.797
MASS:10[illegible] PER CENT : +1.178
MASS:115 PER CENT : +52.025

MEASUREMENT CONDITIONS

DATE 2/10/83 45 mA
SAMPLE REFERENCE: U603 60u

C-3 (cont'd)

```
MASS:                64
MASS:               107
MASS:               115

MASS REFERENCE:      64
                ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME:    5
WAITING TIME :       0
NUMBER OF CYCLES:    5
NUMBER OF BLOCKS:    1
```

RESULTS

BLOCK No: 1

CYCLE No: 1

64 (REF.-->)	+1.000E+00	CNTS-->1.0458E+07
107/REF.-->	+8.829E-03	CNTS-->1.6646E+05
115/REF.-->	+3.497E-01	CNTS-->3.7011E+06
		BKGD-->7.478E+04

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.1874E+07
107/REF.-->	+1.809E-02	CNTS-->2.0326E+05
115/REF.-->	+2.964E-01	CNTS-->3.5723E+06
		BKGD-->7.478E+04

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.1927E+07
107/REF.-->	+1.208E-02	CNTS-->2.1791E+05
115/REF.-->	+2.964E-01	CNTS-->3.5883E+06
		BKGD-->7.478E+04

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.1984E+07
107/REF.-->	+1.252E-02	CNTS-->2.2387E+05
115/REF.-->	+2.963E-01	CNTS-->3.6058E+06
		BKGD-->7.478E+04

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.2012E+07
107/REF.-->	+1.280E-02	CNTS-->2.2760E+05
115/REF.-->	+2.940E-01	CNTS-->3.5843E+06
		BKGD-->7.478E+04

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+1.142E-02
MASS:115	MEAN:	+3.065E-01

ATOMIC o/o

MASS: 64	PER CENT	:	+75.876
MASS:107	PER CENT	:	+.867
MASS:115	PER CENT	:	+23.257

WEIGHT (o/o)

MASS: 64	PER CENT	:	+63.699
MASS:107	PER CENT	:	+1.217
MASS:115	PER CENT	:	+35.084

MEASUREMENT CONDITIONS

DATE: 2/10/83

SAMPLE REFERENCE: U603 RA578 19mA

PRIMARY IONS: OXYGEN

SECONDARY IONS:

C-3 (cont'd)

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.5786E+07
107/REF.-->	+1.202E-04	CNTS-->3.8830E+03
115/REF.-->	+4.935E-04	CNTS-->9.7740E+03
		BKGD-->1.985E+03

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.5934E+07
107/REF.-->	+1.153E-04	CNTS-->3.7710E+03
115/REF.-->	+4.886E-04	CNTS-->9.7180E+03
		BKGD-->1.934E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.5981E+07
107/REF.-->	+1.098E-04	CNTS-->3.6600E+03
115/REF.-->	+4.922E-04	CNTS-->9.7410E+03
		BKGD-->1.915E+03

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.5999E+07
107/REF.-->	+1.061E-04	CNTS-->3.5760E+03
115/REF.-->	+4.930E-04	CNTS-->9.7470E+03
		BKGD-->1.860E+03

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.6042E+07
107/REF.-->	+1.079E-04	CNTS-->3.5230E+03
115/REF.-->	+4.902E-04	CNTS-->9.7850E+03
		BKGD-->1.793E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+1.118E-04
MASS:115	MEAN:	+4.931E-04

ATOMIC o/o

MASS: 64	PER CENT	:	+99.940
MASS:107	PER CENT	:	+.011
MASS:115	PER CENT	:	+.049

WEIGHT (o/o)

MASS: 64	PER CENT	:	+99.893
MASS:107	PER CENT	:	+.019
MASS:115	PER CENT	:	+.089

MEASUREMENT CONDITIONS

DATE 2/10/83 51 nA
SAMPLE REFERENCE: U605 150

Test Run 1

C-3 (cont.)

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

Calib. Points

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.2559E+07
107/REF.-->	+1.302E-04	CNTS-->4.0670E+03
115/REF.-->	+4.881E-04	CNTS-->6.4610E+03
		BKGD-->2.332E+03

60%

CYCLE No: 2

64/REF.-->	1.000E+00	CNTS-->1.3444E+07
107/REF.-->	+9.410E-05	CNTS-->2.7660E+03
115/REF.-->	+4.997E-04	CNTS-->8.2180E+03
		BKGD-->1.501E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.3441E+07
107/REF.-->	+8.1[illegible]	CNTS-->2.[illegible]
[illegible]	[illegible]	
		BKGD-->1.148E+03

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.3503E+07
107/REF.-->	+6.695E-05	CNTS-->1.8920E+03
115/REF.-->	+4.329E-04	CNTS-->6.8330E+03
		BKGD-->9.880E+02

0.01% Ag

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.3581E+07
107/REF.-->	+5.854E-05	CNTS-->1.6620E+03
115/REF.-->	+4.203E-04	CNTS-->6.5750E+03
		BKGD-->8.670E+02

0.01% In

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+8.779E-05
MASS:115	MEAN:	+4.569E-04

ATOMIC o/o

MASS: 64	PER CENT :	+99.946
MASS:107	PER CENT :	+.089
MASS:115	PER CENT :	+.046

HEIGHT (o/o)

MASS: 64	PER CENT :	+99.903
MASS:107	PER CENT :	+.015
MASS:115	PER CENT :	+.082

MEASUREMENT CONDITIONS 33nA : lower transfer syst.

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

C-4

May 29, 1984

Dr. Louis J. Cabri
Canada Centre for Mineral &
Energy Technology
Canada Dept. of Energy Mines &
Resources
555 Booth Street
Ottawa, Ontario M1A 0G1

Dear Louis:

I wish to summarize what we discussed a week ago when you visited London.

1. The last series of experiments on block WA used an O^- beam of some 5 times higher intensity than in the experiments described in the Interim Report. The calibration graph (Figure 1) was identical to earlier graphs (see Figure 2 in the Interim Report).

2. Specimen 8 was analyzed under the new conditions and the results compared with earlier data obtained with the smaller (and less intense) ion beam. The crater produced by the stronger beam (see #3 in Figure 2) is about 2.5 times larger than #1 and #2, produced previously. Note that one can't exactly equate beam ion size and crater size, since the crater enlarges with extended sputtering. An enlarged view of hole #3 is seen in Figure 3. A small amount of beam-induced roughening appears at the crater bottom. A profilometer trace across the holes shows that hole #3 (2500 A°) is about four times deeper than holes 1 and 2. Since the analysis period used in the recent series is twice that of the earlier runs, it appears that the sputter rate is at least twice as fast with the larger beam (my guess is about 125 A°/min).

3. The silver and indium data is given for 8A-8F in Table 1 and is compared with earlier runs. As indicated earlier we ran two blocks of analyses here, rather than one as we had done for the Interim Report. It appears that the concentration values flatten out, usually during the second data series. The drop off in concentration is not as drastic as earlier since the higher intensity beam has penetrated further before the analyses was actually begun. Note that there appears to be some relationship between Ag and In concentrations. In summary, then, the earlier

C-4 (cond)

results were definitly distorted by surface enrichment. It is necessary to penetrate ~0.2 um before this enrichment is removed.

4. It seems to me that we should continue to use the higher intensity beam in the future and try to achieve microscopic analysis by depth profile measurements in the z direction. Unfortunately one can't correlate this against any feature on the surface, but it's the only trace analysis procedure I know with sub-micron resolution.

We're set to try the galenas at the next session. We will be looking carefully at the standards first.

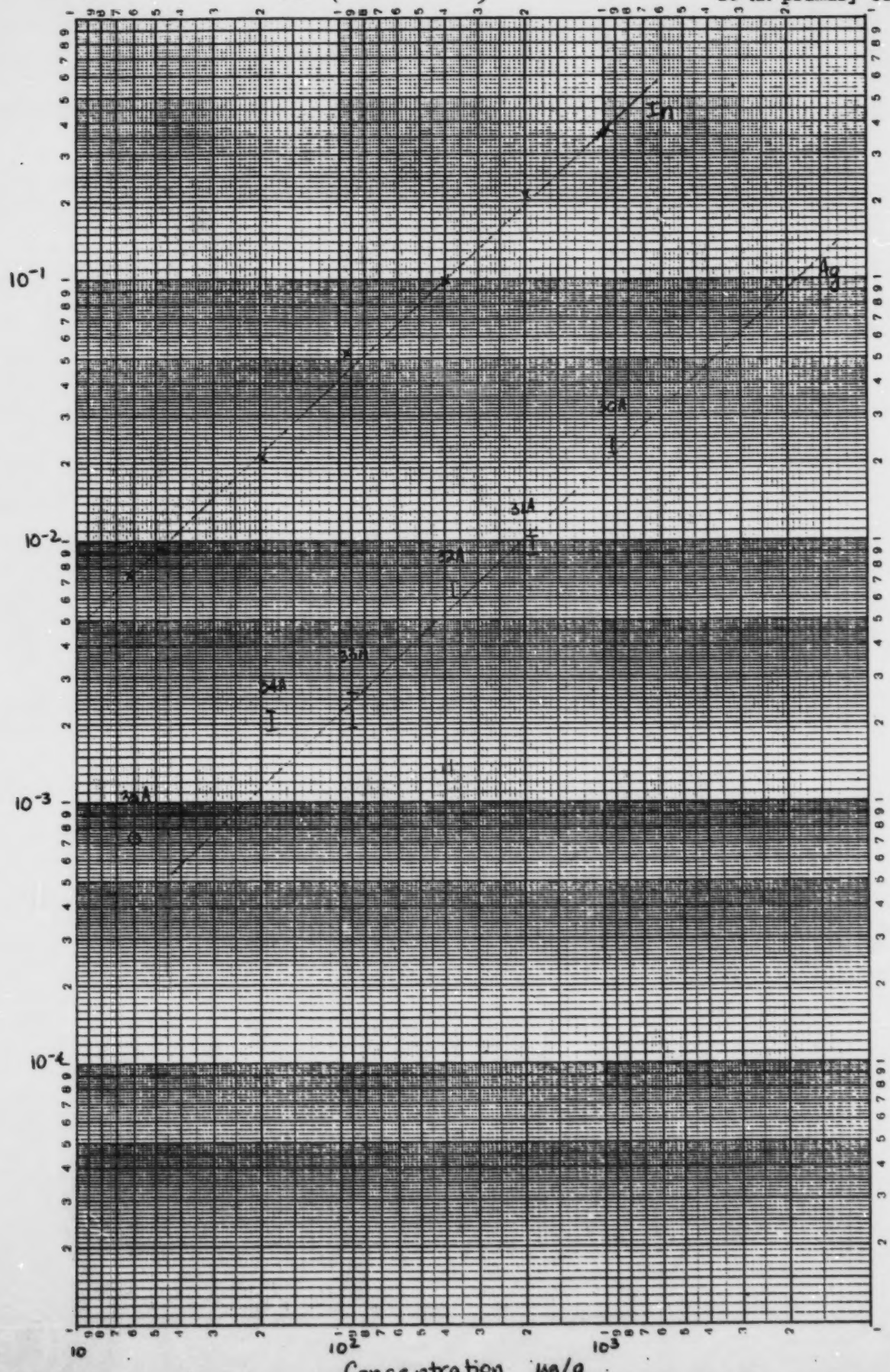
Sorry for the rushed and probably confused letter.

Best reagards
yours very truly

N. S. Mc Intyre

c.c. W. J. Chauvin

Figure 1 C-4 (cont) May 9 Run WA Stds
20 nA primary current



X/Zn

GRAPHIC CONTROLS CANADA LTD. MADE IN CANADA

G-125 LOGARITHMIC 5 X 3 CYCLES SPECIFY TRACING OR DRAWING PAPER

C-4-((cont)

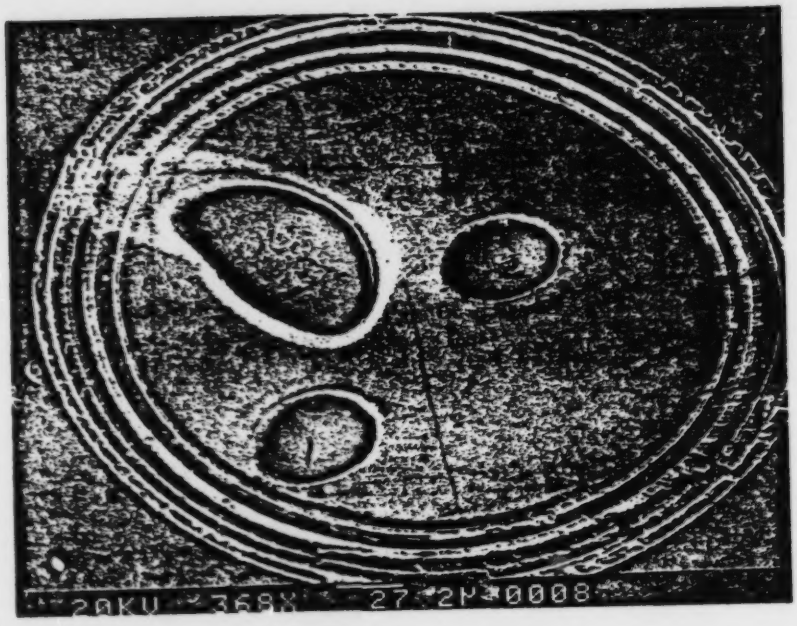


Figure 2

SEM of Specimen 8A

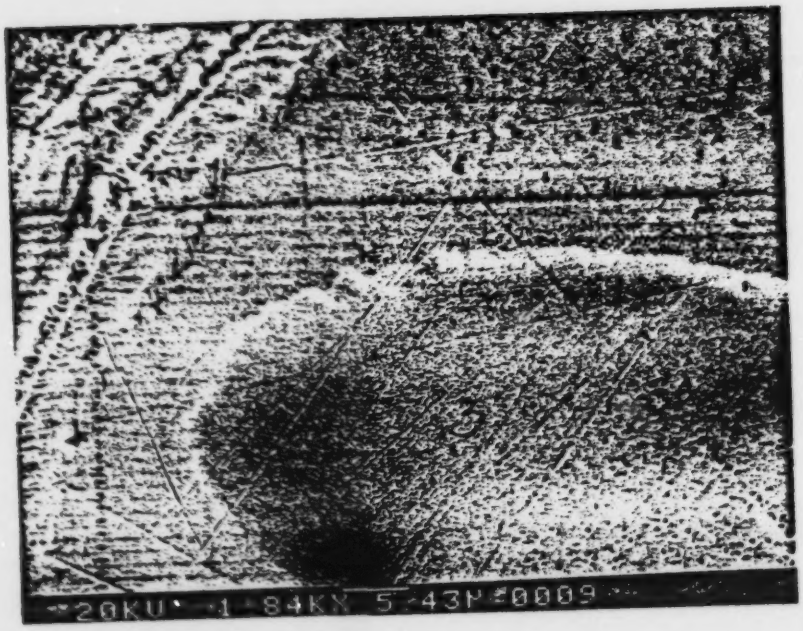


Figure 3

Enlarged SEM of hole #3 above

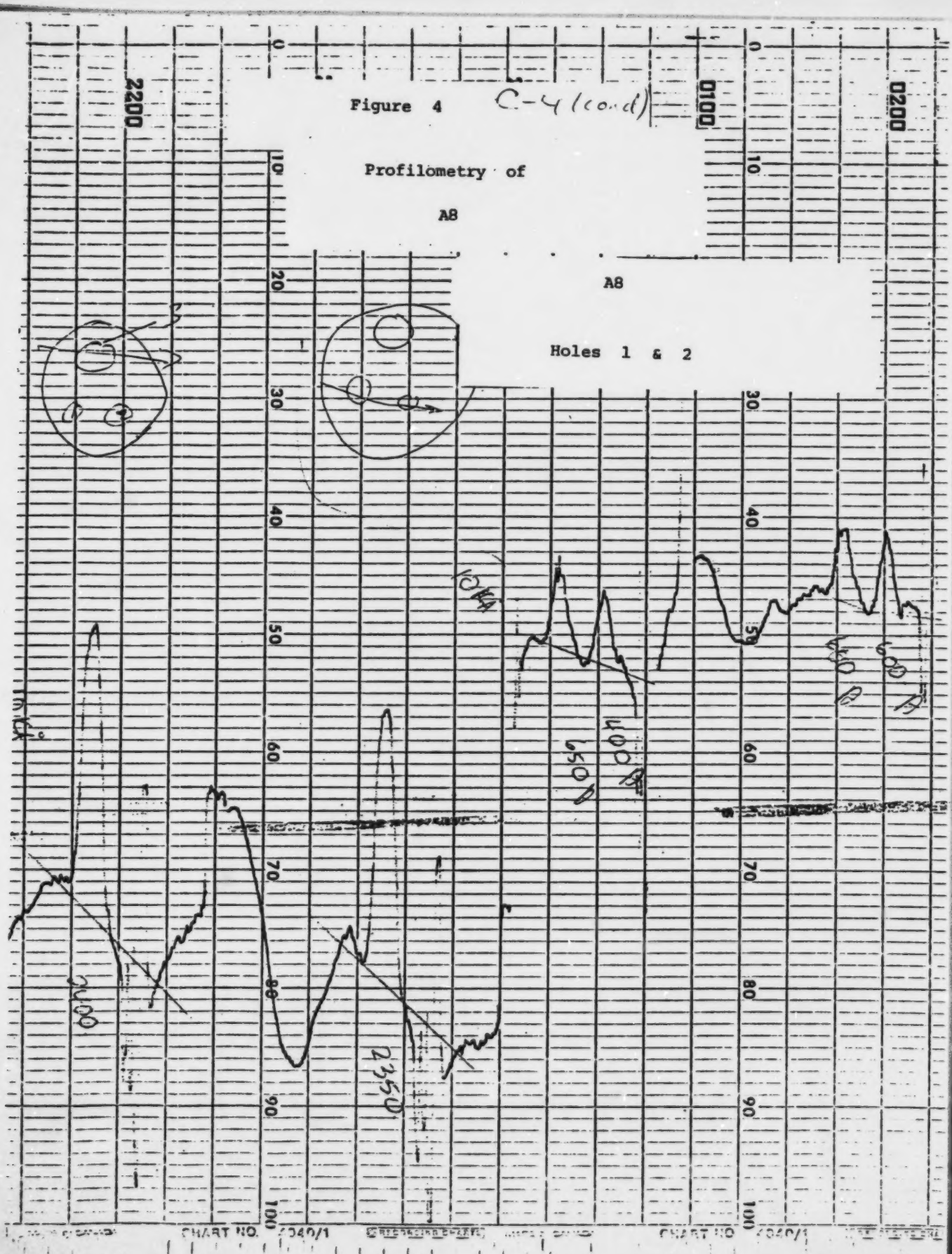

C-4 (cont'd)
Profilometry of
A8
A8
Holes 1 & 2
2200
0100
0200
CHART NO. 1040/1
CHART NO. 1040/1

Figure 4

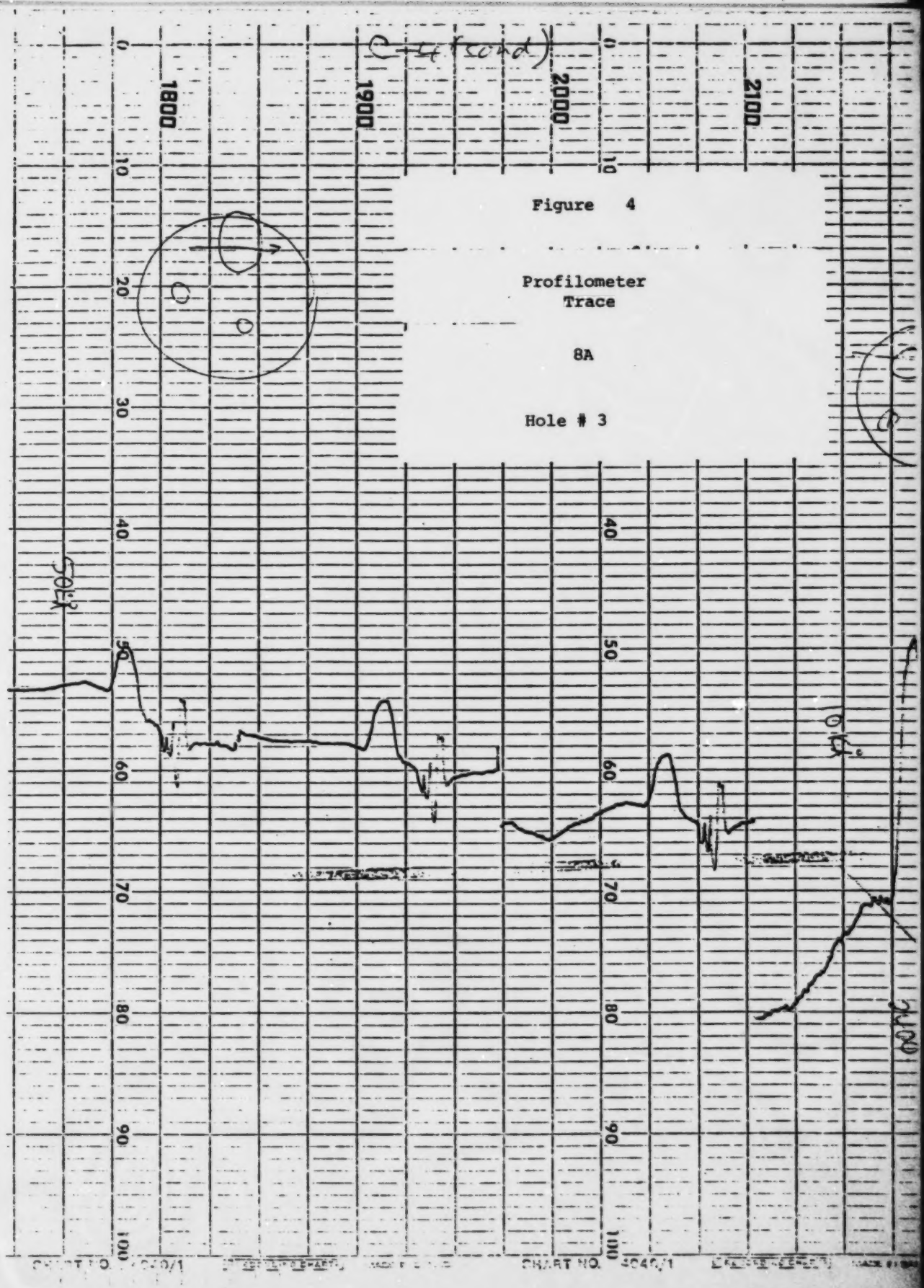


Figure 4

Profilometer
Trace

8A

Hole # 3

C-4 (cont'd)

Table 1 - Silver & Indium Results

	May 9 Run	Previous Runs	(Lower Current)
8A-3	~~9.30~~ 730	8A-1 → 10,640	8A-2 → 1,505
	~~8.50~~ 850	↓	↓
	~~8.05~~ 805		
	~~7.80~~ 780		
Ag	803	8,620	1,080
	850		
	850		
	880		
	900		
8A-3	4.1	8A-1 63	14
	3.4		
	3.4	↓	↓
	3.0		
	2.6	56	11
In			
	2.6		
	2.6		
	2.6		
	2.6		
	2.6		
8B-3	750	8B-1 1,265	1,170
	670		
	550	↓	↓
	555		
	560	1,002	767
Ag			
	530		
	530		
	530		
	530		
	530		
8B-3	4.0	8B-1 13	13
	3.5		
	3.2	↓	↓
	3.0		
	2.0	11	10
In			
	2.6		
	2.6		
	2.6		
	2.6		
	2.6		

C-41(cont'd)

Table 1 Cont...

	May 9 Run	Previous Runs	
8C-3	2400	8C1 880	8C2 1,010
	2700		
	2800	↓	↓
	2900		
	2900	540	560
Ag			
	3000.		
	2900.		
	2700.		
	2600.		
	2600.		
8C-3	17.0	11	12
	18.5		
In	19.2	↓	↓
	19.2		
	19.2	8	8
	19.0		
	20.0		
	18.0		
	18.0		
	19.0		
8D-3	1350	760	1,300
	1260		
	1220	↓	↓
	1150		
	1200	396	900
Ag			
	1220		
	1250		
	1250		
	1300		
	1325		
	3.8	12	9
	3.3		
In	3.1	↓	↓
	3.0		
	2.9	8	6
	2.6		
	2.6		
	2.6		
	2.6		
	2.6		

C-4 (cont).

TABLE 1 Cont...

	May 9 Run	Previous Runs			
8E-3	3750	8E-1	1,005	8E-2	1,560
	3300				
	3000		↓		↓
	3000				
	2850				
Ag	2750		761		1,020
	2600				
	2500				
	2650				
	2800				
	2650				
In	32.0		9		11
	28.3				
	26.0		↓		↓
	24.5				
	23.0		7		7
	20.0				
	19.0				
	18.0				
	19.0				
	17.0				
8F-3	5900		700		780
	5850				
	5900				
	5850				
	6000				
Ag	5900		↓		↓
	5950				
	6000				
	6500				
	6300		327		450
	64.0				
	50.0				
	47.0				
	40.0				
	38.0				
In	33.0				
	33.0				
	29.0				
	31.0				
	29.0				
	28.0				

C-5

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

Research and Technology
Recherche et technologie

Canada Centre for Mineral and Energy Technology,
555 Booth Street
Ottawa, Ontario
K1A 0G1

Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa (Ontario)
K1A 0G1

Your file Votre référence

Our file Notre référence

June 12, 1984 BY COURIER

C-5

Dr. N.S. McIntyre,
Technical Director,
Surface Science,
Western Centre for Interdisciplinary
Studies in Chemical Physics,
University of Western Ontario,
London, Ontario
N6A 3K7

Dear Stewart:

Thank you for your letter (postmarked 29 May 1984) with the results of the May 9 run and other comments. I have some questions and comments.

1. You ran "two blocks of analyses" on May 9. What does that mean exactly? 2 sets of 5 cycles on the same area (without moving off or moving off and back again?) or 2 sets of 5 cycles on different parts of the same circled area?
2. 8E-3 and 8F-3 seem to have been done for one set of 11 cycles?
3. While I agree that the results appear to flatten out, there is still enough variability to cause a good deal of worry. Also these variations go both ways.
4. As I had discussed with you in London, Sample 8 is not a partially good candidate for this work and I suggest you try just 15 analyses on a new section that I enclose (Block B sphalerite). There are 15 sphalerite grains circled with the numbers 1,3,4,4A,5,7,10,13,14,18,19,20,22,24 and 25. Please be sure that the beam is placed dead centre in every case.

I only have two useful standards in this section, Nos. 598 and 622, but I thought you could establish your standard curve from Block A (23/8/83) and use 598 and 622 as checks -

598	0.114% Ag	0.119% In
622	0.073% Ag	0.079% In.

5. Please also refer to my letter of 6 April when I asked that you analyse 6, 8, and 10 in the section A of 25/10/83.

If there is anything unclear, please phone.

Kindest regards,

Louis J. Cabri

LJC/tj

Canada

C-6
Data sent to L. Cabri
June 29, 1984.

C-6
Sent to L. Cabri
June 29, 1984.

Table II

Analysis of Natural Sphalerites for Silver and Indium

Specimen	Time into Analysis* (sec)	Silver Concentration (ug/g)	Indium Concentration (ug/g)
USP-1 (Area 1)	30	40	0.9
	60	35	0.8
	90	35	0.8
	120	35	0.7
	150	36	0.7
USP-2 (Area 1)	30	51	0.8
	60	48	0.8
	90	49	0.7
	120	48	0.6
	150	52	0.6
USP-2 (Area 2)	30	1400	0.8
	60	1477	0.7
	90	1516	0.6
	120	1563	0.6
	150	1593	0.6
USP-3 (Area 1)	30	81	0.8
	60	86	0.6
	90	88	0.6
	120	93	0.6
	150	97	0.5

* Sputtering rate of sphalerite
Under these beam conditions is 0.6 - 1.0 nm/sec

Data

C-6 (cont)

Zinc Sphalerite
Unknowns.

64
107
115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT

MEASUREMENT TIME: 5
WAITING TIME : 8
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.4055E+07
107/REF.-->	+6.830E-04	CNTS-->1.9005E+04
115/REF.-->	+3.088E-04	CNTS-->1.1862E+04
		BKGD-->5.968E+03

CYCLE No: 2

64 REF.-->	+1.000E+00	CNTS-->2.0807E+07
107 REF.-->	+6.726E-04	CNTS-->1.9457E+04
115/REF.-->	+2.773E-04	CNTS-->1.1233E+04
		BKGD-->5.465E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->2.1243E+07
107/REF.-->	+7.251E-04	CNTS-->2.0864E+04
115/REF.-->	+2.614E-04	CNTS--> 1.0996E+04
		BKGD-->5.465E+03

CYCLE No: 4

64/RE	+1.000E+00	CNTS--> 2.1352E+07
	+7.[illegible]E-04	CNTS--> 2.5321E+04
[illegible]	[illegible]E-04	CNTS--> 1.0674E+04
		BKGD-->5.248E+03

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->2.1470E+07
107/REF.-->	+9.502E-04	CNTS-->2.5166E+04
115 REF.-->	+2.483E-04	CNTS-->1.0540E+04
		BKGD--> 5.200E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+7.502E-04
MASS:115	MEAN:	+2.699E-04

ATOMIC o o

MASS: 64	PER CENT :	+99.894
MASS:107	PER CENT :	+.079
MASS:115	PER CENT :	+.027

WEIGHT (o/o)

MASS: 64	PER CENT :	+99.820
MASS:107	PER CENT :	+.132
MASS:115	PER CENT :	+.048

ELEMENT CONDITION

REFERENCE: [illegible]PSAI

C-6 (cond)

MEASUREMENT CONDITIONS

MASS:	64
MASS:	107
MASS:	115
MASS REFERENCE:	64

ALL ISOTOPES ARE PRESENT

MEASUREMENT TIME:	5
WAITING TIME :	0
NUMBER OF CYCLES:	5
NUMBER OF BLOCKS:	1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.7084E+07
107/REF.-->	+7.561E-04	CNTS-->1.8052E+04
115/REF.-->	+2.966E-04	CNTS-->1.0204E+04
		BKGD-->5.138E+03

USIP 2F

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.8006E+07
107/REF.-->	+7.184E-04	CNTS-->1.7594E+04
115/REF.-->	+2.650E-04	CNTS-->9.4310E+03
		BKGD-->4.661E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.8346E+07
107/REF.-->	+7.296E-04	CNTS-->1.7811E+04
115/REF.-->	+2.413E-04	CNTS-->8.8540E+03
		BKGD-->4.429E+03

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.8450E+07
107/REF.-->	+7.213E-04	CNTS-->1.7601E+04
115/REF.-->	+2.349E-04	CNTS-->8.6290E+03
		BKGD-->4.296E+03

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS--> 1.8535E+07
107/REF.-->	+7.601E-04	CNTS--> 1.8181E+04
115/REF.-->	+2.278E-04	CNTS--> 8.3170E+03
		BKGD--> 4.096E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+7.371E-04
MASS:115	MEAN:	+2.531E-04

7.4x10⁻⁴

ATOMIC o/o

MASS: 64	PER CENT :	+99.901
MASS:107	PER CENT :	+.074
MASS:115	PER CENT :	+.025

WEIGHT (o/o)

MASS: 64	PER CENT :	+99.832
MASS:107	PER CENT :	+.123
MASS:115	PER CENT :	+.045

MEASUREMENT CONDITIONS

DATE 2-10 83 59 nA C-

C-6 (cont)

MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 8
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	1400	CNTS-->2.0664E+07
107/REF.-->	+3.868E-02		CNTS-->8.0514E+05
115/REF.-->	+2.857E-04	0.8	CNTS-->1.1951E+04
			BKGD-->6.050E+03

CYCLE No: 2

64/REF.-->	+1.000E+00		CNTS-->2.2402E+07
107/REF.-->	+4.075E-02	1477	CNTS-->9.1829E+05
115/REF.-->	+2.504E-04	0.7	CNTS-->1.1260E+04
			BKGD-->5.652E+03

CYCLE No: 3

64/REF.-->	+1.000E+00		CNTS-->2.3125E+07
107/REF.-->	+4.182E-02	1516	CNTS-->9.7258E+05
115/REF.-->	+2.361E-04	0.6	CNTS-->1.1110E+04
			BKGD-->5.632E+03

CYCLE No: 4

64/REF.-->	+1.000E+00		CNTS-->2.3438E+07
107/REF.-->	+4.313E-02	1563	CNTS-->1.0160E+06
115/REF.-->	+2.372E-04	0.6	CNTS-->1.1064E+04
			BKGD-->5.505E+03

CYCLE No: 5

64/REF.-->	+1.000E+00		CNTS-->2.3705E+07
107/REF.-->	+4.395E-02	1573	CNTS-->1.0470E+06
115/REF.-->	+2.220E-04	0.6	CNTS-->1.0580E+04
			BKGD-->5.319E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+4.167E-02
MASS:115	MEAN:	+2.463E-04

ATOMIC o/o

MASS: 64	PER CENT	:	+95.977
MASS:107	PER CENT	:	+3.999
MASS:115	PER CENT	:	+.024

WEIGHT (o/o)

MASS: 64	PER CENT	:	+93.449
MASS:107	PER CENT	:	+6.510
MASS:115	PER CENT	:	+.041

MEASUREMENT CONDITIONS

DATE 2/10/83
SAMPLE REFERENCE: USP3A2

C to (cont)

DATE	3/10/83
SAMPLE REFERENCE:	USP2A2
PRIMARY IONS:	OXYGEN
SECONDARY IONS:	
PRIMARY CURRENT:	70NANO AMP
HIGH VOLTAGE:	11.9KILOVOLTS
RASTERING :	0 MICRONS
TRANSFER OPTICS :	150 MICRONS

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00		CNTS-->1.6482E+07
107/REF.-->	+5.483E-04	40 ug/g	CNTS-->1.4128E+04
115/REF.-->	+3.127E-04	0.9 ug/g	CNTS-->1.0245E+04
			BKGD-->5.893E+03

CYCLE No: 2

64/REF.-->	+1.000E+00		CNTS-->1.7493E+07
107/REF.-->	+4.816E-04	35	CNTS-->1.3285E+04
115/REF.-->	+2.851E-04	0.8	CNTS-->9.8490E+03
			BKGD-->4.863E+03

CYCLE No: 3

64/REF.-->	+1.000E+00		CNTS-->1.7868E+07
107/REF.-->	+4.900E-04	35	CNTS-->1.3153E+04
115/REF.-->	+2.718E-04	0.8	CNTS-->9.4330E+03
			BKGD-->4.578E+03

CYCLE No: 4

64/REF.-->	+1.000E+00		CNTS-->1.7977E+07
107/REF.-->	+4.769E-04	35	CNTS-->1.3007E+04
115/REF.-->	+2.517E-04	0.7	CNTS-->8.9600E+03
			BKGD-->4.436E+03

CYCLE No: 5

64/REF.-->	+1.000E+00		CNTS-->1.8016E+07
107/REF.-->	+5.012E-04	36	CNTS-->1.3282E+04
115/REF.-->	+2.547E-04	0.7	CNTS-->8.8430E+03
			BKGD-->4.255E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+4.976E-04
MASS:115	MEAN:	+2.752E-04

ATOMIC o/o

MASS: 64	PER CENT	:	+99.923
MASS:107	PER CENT	:	+.050
MASS:115	PER CENT	:	+.027

WEIGHT (o/o)

59 nA

MASS: 64	PER CENT	:	+99.868
MASS:107	PER CENT	:	+.061

C-6 (cont'd)

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

Calibration

RESULTS

Calib Point (

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->8.3562E+06
107/REF.-->	+1.405E-02	CNTS-->2.2709E+05
115/REF.-->	+5.994E-01	CNTS-->5.0533E+06
		BKGD-->1.113E+05

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->8.5205E+06
107/REF.-->	+1.436E-02	CNTS-->2.3222E+05
115/REF.-->	+6.008E-01	CNTS-->5.1633E+06
		BKGD-->1.113E+05

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->8.5196E+06
107/REF.-->	+1.544E-02	CNTS-->2.4107E+05
115/REF.-->	+6.172E-01	CNTS-->5.3006E+06
		BKGD-->1.113E+05

0.1 % Ag
0.1 % In

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->8.509[illegible]
107/REF.-->	+1.538E-02	CNTS-->2.404[illegible]
115/REF.-->	+6.306E-01	CNTS-->5.4089E+06
		BKGD-->1.113E+05

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->8.4539E+06
107/REF.-->	+1.602E-02	CNTS-->2.4491E+05
115/REF.-->	+6.454E-01	CNTS-->5.4968E+06
		BKGD-->1.113E+05

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+1.505E-02
MASS:115	MEAN:	+6.187E-01

ATOMIC o/o

MASS: 64	PER CENT	:	+61.209
MASS:107	PER CENT	:	+.921
MASS:115	PER CENT	:	+37.870

WEIGHT (o/o)

MASS: 6	PER CENT	:	+46.797
MASS:1[illegible]	PER CENT	:	+1.178
MASS:11[illegible]	PER CENT	:	+52.025

MEASUREMENT CONDITIONS

DATE 2/10/83
FILE REFERENCE: U603 60u

45 nA

C-6 (cont'd)

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64 REF.-->	+1.000E+00	CNTS-->1.0458E+07
107/RE.-->	+8.829E-03	CNTS-->1.6646E+05
115/RE.-->	+3.497E-01	CNTS-->3.7011E+06
		BKGD-->7.478E+04

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.1874E+07
107/REF.-->	+1.089E-02	CNTS-->2.0326E+05
115/REF.-->	+2.964E-01	CNTS-->3.5723E+06
		BKGD-->7.478E+04

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.1927E+07
107/REF.-->	+1.208E-02	CNTS-->2.1791E+05
115/REF.-->	+2.964E-01	CNTS-->3.5883E+06
		BKGD-->7.478E+04

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.1984E+07
107/REF.-->	+1.252E-02	CNTS-->2.2387E+05
115/REF.-->	+2.965E-01	CNTS-->3.6058E+06
		BKGD-->7.478E+04

CYCLE No: 5

64/REF.-->	+1.000E+00	CNTS-->1.2012E+07
107/REF.-->	+1.268E-02	CNTS-->2.2760E+05
115/REF.-->	+2.940E-01	CNTS-->3.5643E+06
		BKGD-->7.478E+04

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64	MEAN:	+1.000E+00
MASS:107	MEAN:	+1.142E-02
MASS:115	MEAN:	+3.065E-01

ATOMIC o/o

MASS: 64	PER CENT	: +75.876
MASS:107	PER CENT	: +.867
MASS:115	PER CENT	: +23.257

WEIGHT (o/o)

MASS: 64	PER CENT	: +63.699
MASS:107	PER CENT	: +1.217
MASS:115	PER CENT	: +35.084

MEASUREMENT CONDITIONS

DATE 2/10 03
SAMPLE REFERENCE: U603 PA570 1911A
PRIMARY IONS: OXYGEN

C-6 (cond)

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

MASS REFERENCE: 64
ALL ISOTOPES ARE PRESENT
MEASUREMENT TIME: 5
WAITING TIME : 8
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

RESULTS

BLOCK No: 1

CYCLE No: 1

64/REF.-->	+1.000E+00	CNTS-->1.5786E+07
107/REF.-->	+1.202E-04	CNTS-->3.8830E+03
115/REF.-->	+4.935E-04	CNTS-->9.7740E+03
		BKGD-->1.985E+03

CYCLE No: 2

64/REF.-->	+1.000E+00	CNTS-->1.5934E+07
107/REF.-->	+1.153E-04	CNTS-->3.7710E+03
115/REF.-->	+4.886E-04	CNTS-->9.7180E+03
		BKGD-->1.934E+03

CYCLE No: 3

64/REF.-->	+1.000E+00	CNTS-->1.5901E+07
107/REF.-->	+1.090E-04	CNTS-->3.6600E+03
115/REF.-->	+4.922E-04	CNTS-->9.7410E+03
		BKGD-->1.915E+03

CYCLE No: 4

64/REF.-->	+1.000E+00	CNTS-->1.5999E+07
107/REF.-->	+1.061E-04	CNTS-->3.570E+03
115/REF.-->	+4.930E-04	CNTS-->9.7470E+03
		BKGD-->1.860E+03

CYCLE No: 5.

64/REF.-->	+1.000E+00	CNTS-->1.6042E+07
107/REF.-->	+1.079E-04	CNTS-->3.5230E+03
115/REF.-->	+4.982E-04	CNTS-->9.7850E+03
		BKGD-->1.793E+03

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64 MEAN:	+1.000E+00
MASS:107 MEAN:	+1.118E-04
MASS:115 MEAN:	+4.931E-04

ATOMIC o/o

MASS: 64	PER CENT :	+99.940
MASS:107	PER CENT :	+.011
MASS:115	PER CENT :	+.049

WEIGHT (o/o)

MASS: 64	PER CENT :	+99.893
MASS:107	PER CENT :	+.019
MASS:115	PER CENT :	+.089

MEASUREMENT CONDITIONS

DATE 2/10/83
SAMPLE REFERENCE: U605 150

51 nA

C-6 (cont).

MEASUREMENT CONDITIONS

MASS: 64
MASS: 107
MASS: 115

REFERENCE: 64

ALL ISOTOPES ARE PRESENT

MEASUREMENT TIME: 5
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 1

Calib Points

RESULTS

BLOCK No: 1

CYCLE No: 1

64 REF.-->
107 REF.-->
115 REF.-->

+1.000E+00
+1.382E-04
+4.881E-04

CNTS-->1.2559E+07
CNTS-->4.0670E+03
CNTS-->6.4610E+03
BKGD-->2.332E+03

CYCLE No: 2

64 REF.-->
107 REF.-->
115 REF.-->

+1.000E+00
+5.410E-05
+4.967E-04

CNTS-->1.3444E+07
CNTS-->2.7660E+03
CNTS-->6.2180E+03
BKGD-->1.501E+03

CYCLE No: 3

64 REF.-->
107 REF.-->
115 REF.-->

+1.000E+00
+8.1[illegible]
+1.4[illegible]

CNTS-->1.[illegible]441E+07
CNTS-->2.[illegible]
[illegible]
BKGD-->1.146E+03

CYCLE No: 4

64/REF.-->
107/REF.-->
115/REF.-->

+1.000E+00
+6.695E-05
+4.329E-04

CNTS-->1.3503E+07
CNTS-->1.8920E+03
CNTS-->6.8330E+03
BKGD-->9.880E+02

0.01% Ag

CYCLE No: 5

64/REF.-->
107/REF.-->
115/REF.-->

+1.000E+00
+5.854E-05
+4.203E-04

CNTS-->1.3581E+07
CNTS-->1.6620E+03
CNTS-->6.5750E+03
BKGD-->8.670E+02

0.01% In.

MEAN RATIO

REFERENCE PEAK MASS: 64

MASS: 64 MEAN: +1.000E+00
MASS:107 MEAN: +8.779E-05
MASS:115 MEAN: +4.569E-04

ATOMIC %

MASS: 64 PER CENT : +99.946
MASS:107 PER CENT : +.009
MASS:115 PER CENT : +.046

WEIGHT (%)

MASS: 64 PER CENT : +99.903
MASS:107 PER CENT : +.015
MASS:115 PER CENT : +.082

MEASUREMENT CONDITIONS

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

C-7

July 12, 1984

Dr. L. J. Cabri
Canada Centre for Mineral &
Energy Technology
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis:

Please find enclosed copies of the raw calibration data taken for the analysis of block WA with a 10um beam during January-March of this year.

We have completed the analysis of Block B sphalerite and will report the results soon. We will next complete the analyses of 6, 8 and 10 in section A of 25/10/83.

Two short runs have been made on block W-C (Galena/Chalcopyrite). Standards 14, 16, 17 and 18 show good reproducibility and are reasonably linear. We've had some reproducibility problems with standards 15 and 19. The natural specimens appear to have the same surface enrichment problems as the sphalerites. We will do some depth profile studies to determine how far the surface should be penetrated before analyses begin.

Yours very truly

N. S. Mc Intyre

C-8

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

C-8

July 16, 1984

Dr. Louis J. Cabri
Canada Centre for Mineral
& Energy Technology
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis:

The SIMS results for 15 analysis on natural sphalerites from block B are enclosed. These were done with a 40um primary beam. The calibration plot used (see Figures 1(a)and (b)) was derived from mean values from all of the block W-A calibration runs. The individual values used, as well as their means, are given in Table 1. The two standards which you provided in block B (598 and 622) served as checks on previous data; these are plotted as red points on the graph in Figure 2, in perspective with the statistical variation in the values obtained on earlier runs. It can be seen that the data from the two block B standards agree well with earlier data.

The complete data on the unknowns is enclosed and is summarized in Table 2. As in the past, we have delayed taking data until it was felt that all signals had stabilized. If the subsequent ion ratios, taken in the first five measurements (block 1), showed less than a ten percent systematic change, the data were accepted. If this was not the case, a second, and even a third block was run. The bracketed values in Table 2 represent the results from the penultimate block of data.

As promised in my letter of May 1, we have run long-term depth profile analyses on some - block B specimens (Figures 3-5). The plots show local variations for sulphur, iron, zinc, silver, cadmium and indium. The crater depths were measured by profilimetry and for two of the profiles, a good crater shape was obtained, indicating that very little roughening of the surface occurs due to the ion beam (see Figure 6). In these cases, we have every reason to expect that the intensity changes measured in the profile represent the actual local variations in concentration. After the intial surface enrichment (quite extreme for indium), variations in silver, indium cadmium are not large on a sub-micron scale.

I assume that you wish us to do more work on block 13, so we have retained it.

Yours very truly,

N. S. Mc Intyre

July 12/04 Mean of std. data.

C-8 (cond)

Fig 1 (a)

DATA

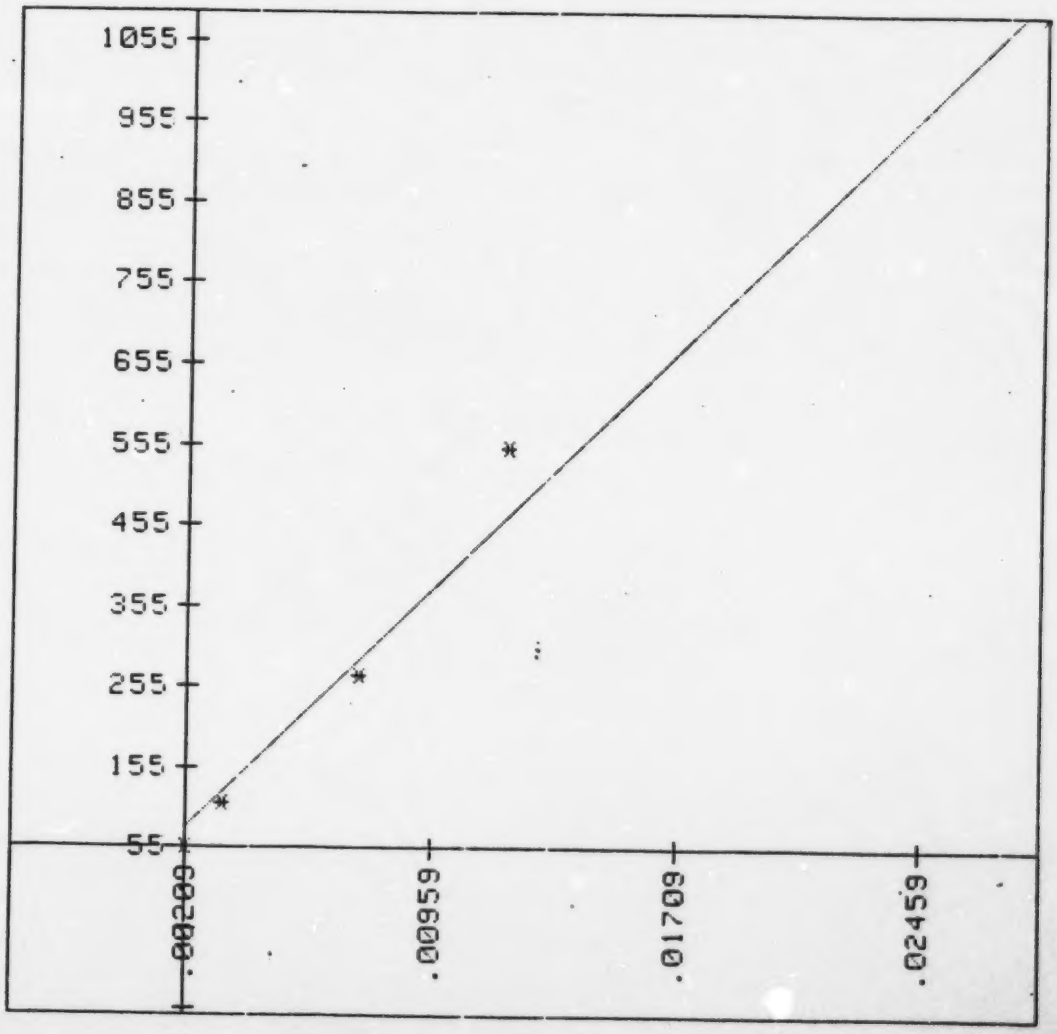
Point #1:	X=.00209	Y=55
Point #2:	X=.00322	Y=110
Point #3:	X=.00737	Y=270
Point #4:	X=.0119	Y=550
Point #5:	X=.0284	Y=1090

Linear model: Y=A+B*X
A=-2.783780324
B= 39428.4428392

Source	Df	SS	MS	F
Regression	1	708661.203	708661.203	240.529
Residual	3	8838.797	2946.266	
Total	4	717500.000		

Ag

LINEAR



C-8 (cond)

DATA

Point #1:	X=.0239	Y=57
Point #2:	X=.0432	Y=110
Point #3:	X=.115	Y=280
Point #4:	X=.237	Y=570
Point #5:	X=.496	Y=1140

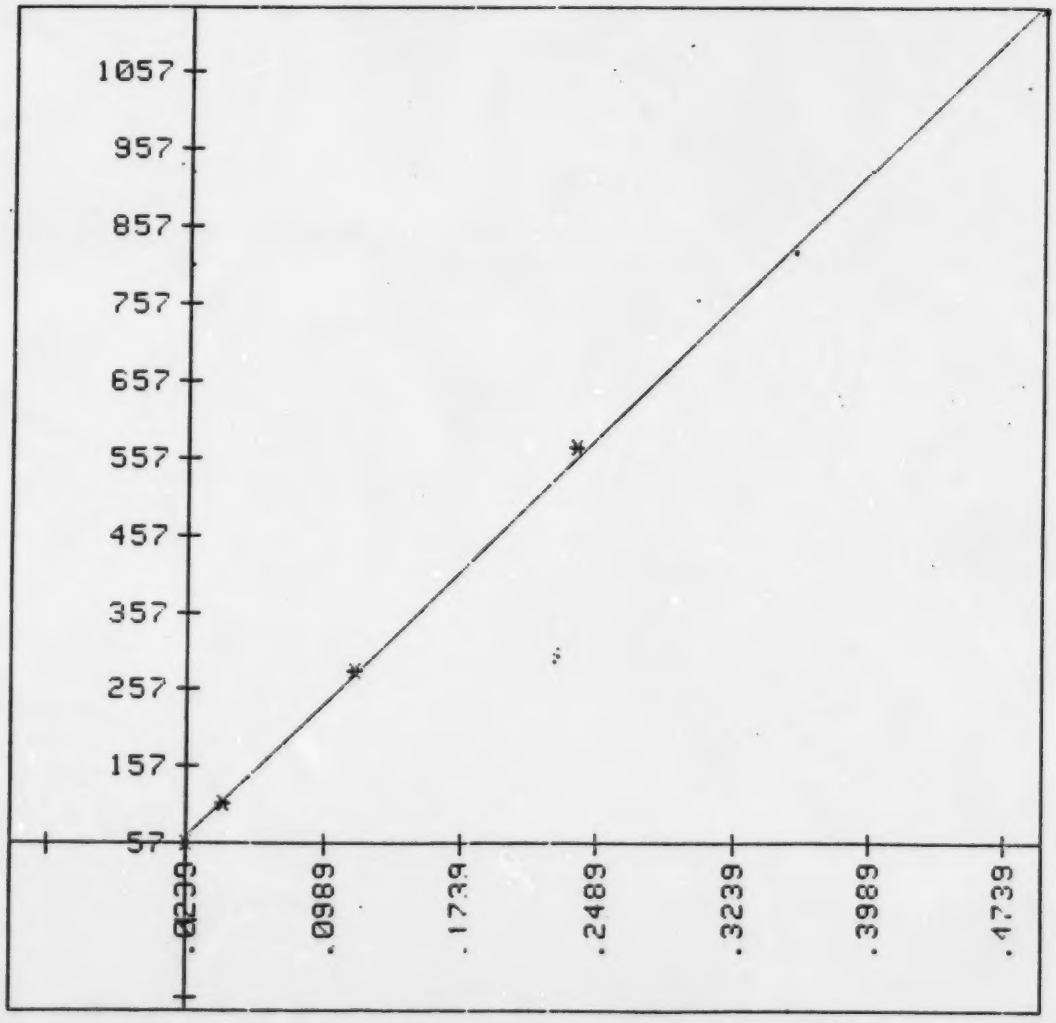
Fig. 2(b)

Linear model: Y=A+B*X
A= 12.609952122
B= 2288.22012828

Source	Df	SS	MS	F
Regression	1	787308.266	787308.266	5747.694
Residual	3	410.934	136.978	
Total	4	787719.200		

In.

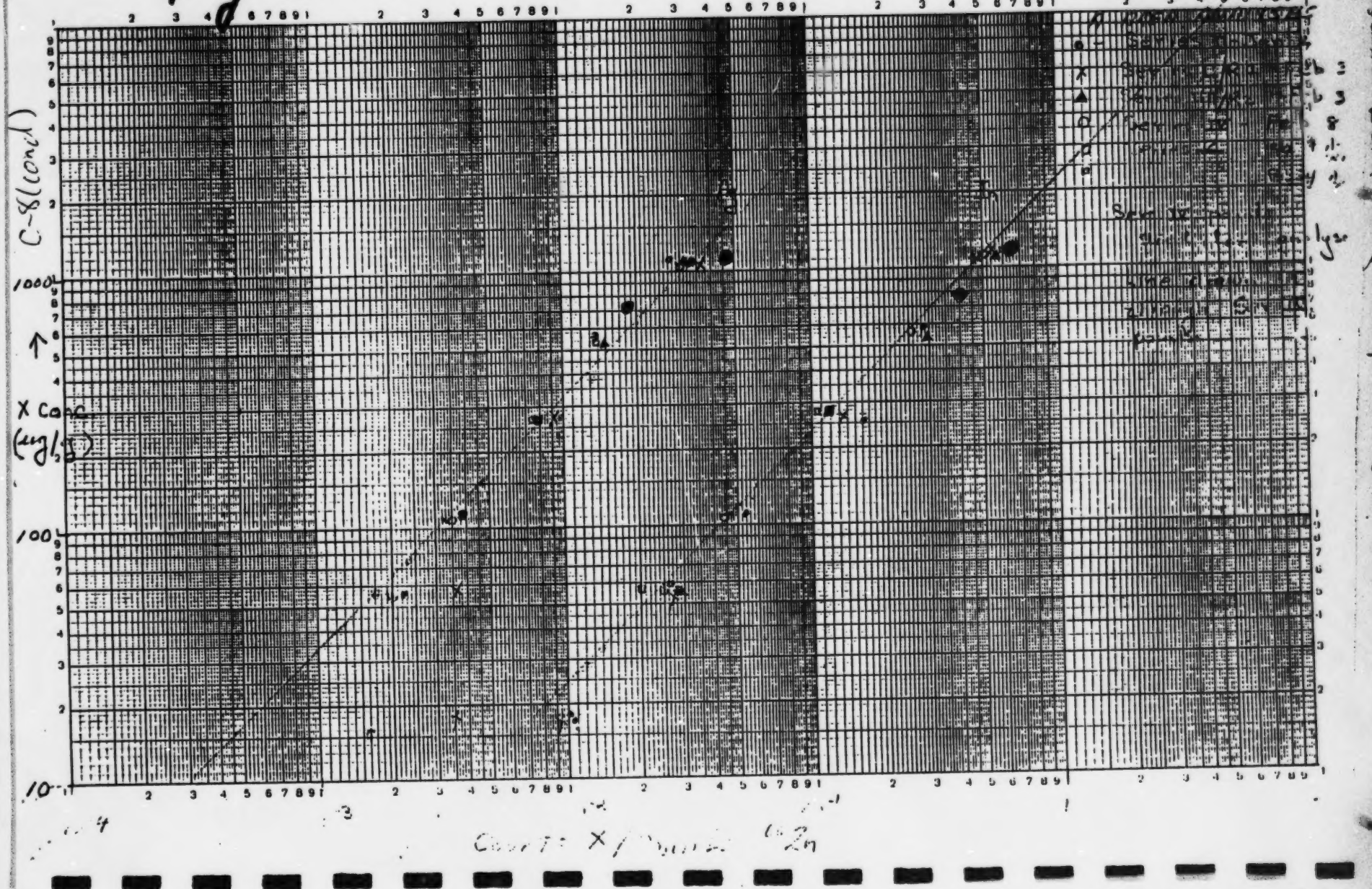
LINEAR



G-125 LOGARITHMIC 5 X 3 CYCLES
SPECIFY TRACING OR DRAWING PAPER

GRAPHIC CONTROLS CANADA LTD.
MADE IN CANADA

Fig 2



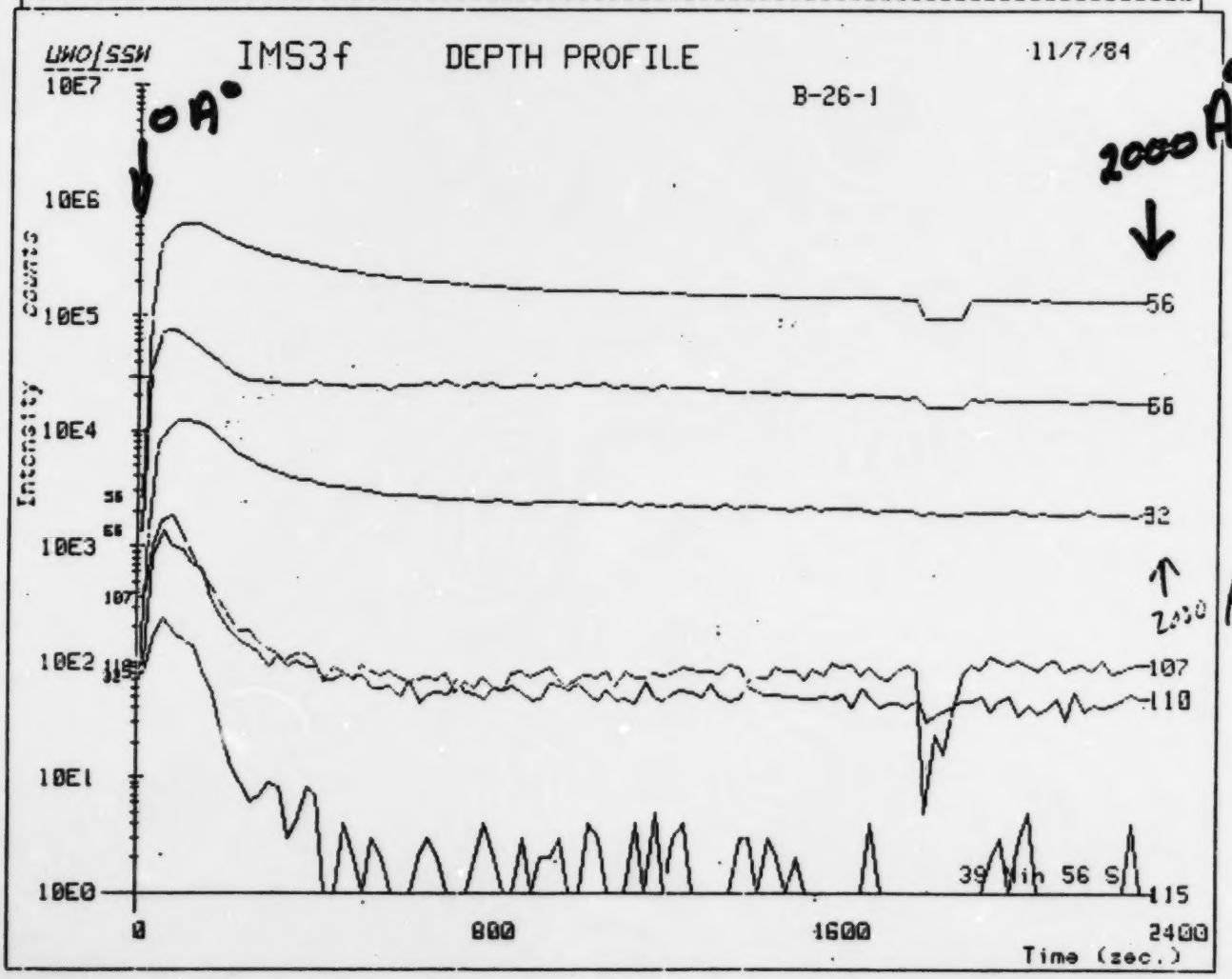
C-8 (cont'd)

Fig 3

MEASUREMENT CONDITIONS

DEAD TIME 129.30E-09

MASS:	32	S
TIME in S./CYCLE :	1.0	
H.V.VOLTAGE:	0.0	
MASS:	56	Fe
TIME in S./CYCLE :	1.0	
H.V.VOLTAGE:	0.0	
MASS:	66	Zn
TIME in S./CYCLE :	1.0	
H.V.VOLTAGE:	0.0	
MASS:	107	Ag
TIME in S./CYCLE :	1.0	
H.V.VOLTAGE:	0.0	
MASS:	110	Cd
TIME in S./CYCLE :	1.0	
H.V.VOLTAGE:	0.0	
MASS:	115	In
TIME in S./CYCLE :	1.0	
H.V.VOLTAGE:	0.0	
TIME PERIOD (Sec):	2400	



SAMPLE: B-26-1
DATE: 11/7/84
PRIMARY IONS : OXYGEN

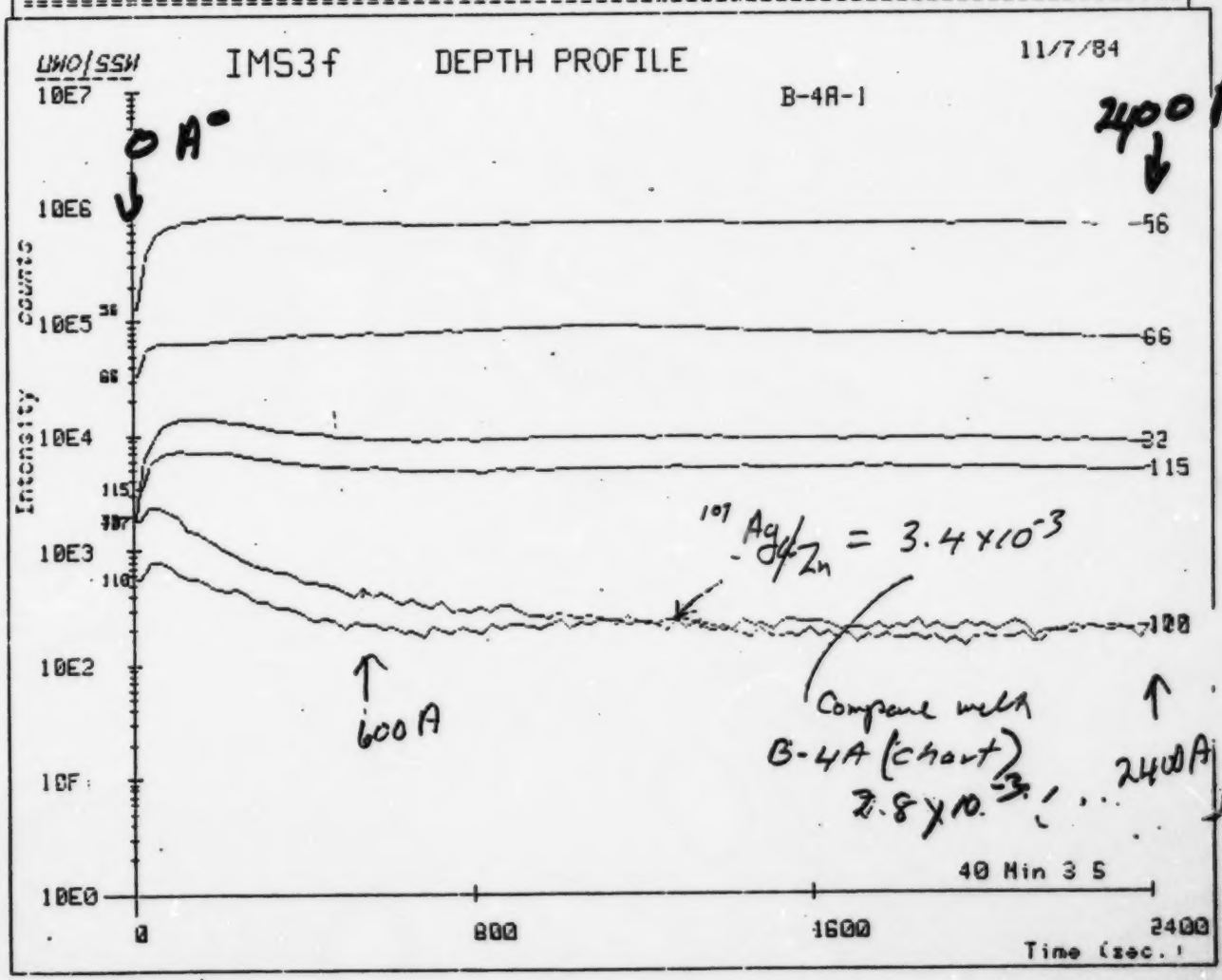
C-8 (cont)

Fig 4

MEASUREMENT CONDITIONS

DEAD TIME 129.30E-09

MASS:	32
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	56
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	66
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	107
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	110
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	115
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
TIME PERIOD (Sec):	2400



SAMPLE: B-4A-1
DATE: 11/7/84
PRIMARY IONS : OXYGEN -

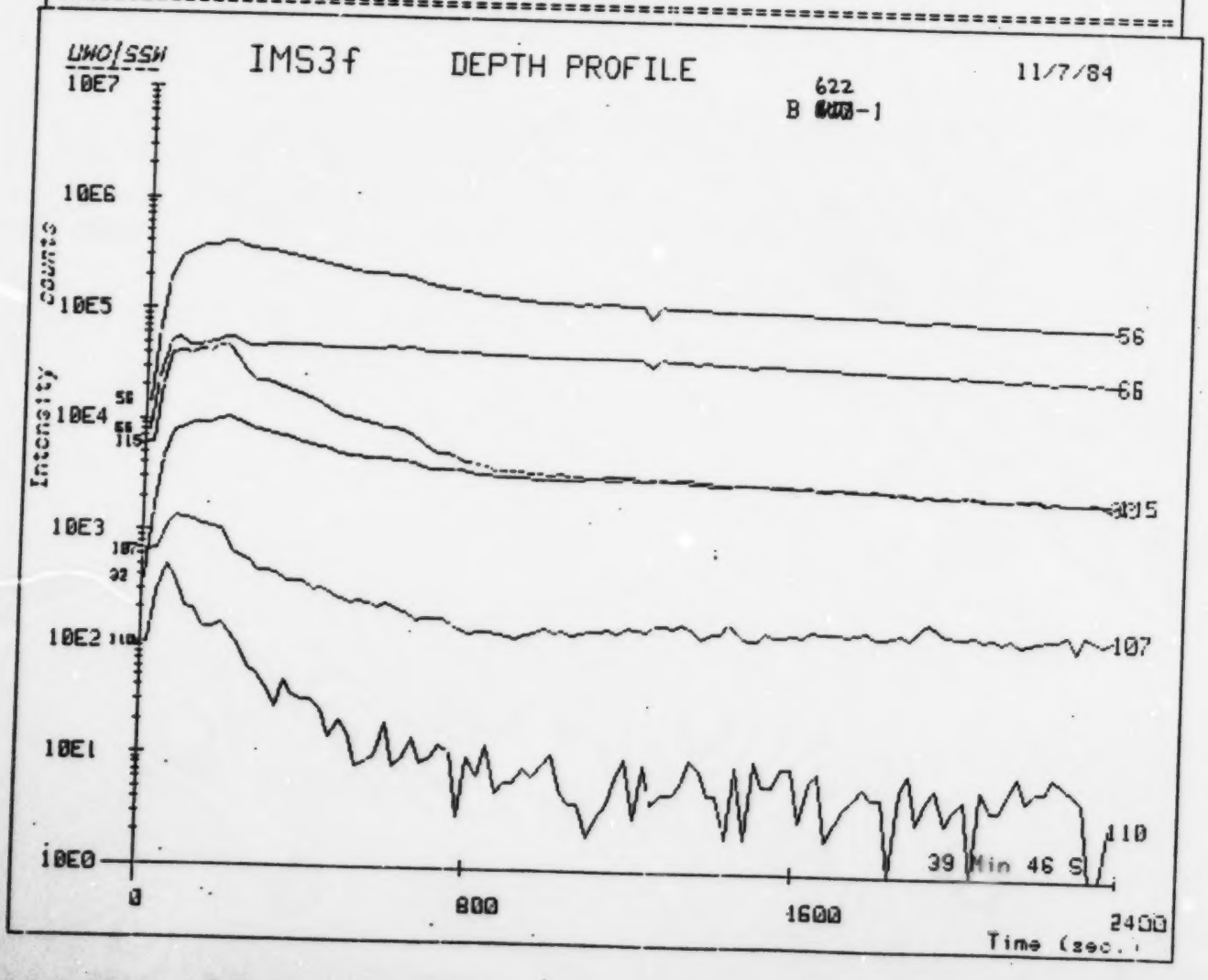
C-8(cond)

Fig 5

MEASUREMENT CONDITIONS

DEAD TIME 129.30E-09

MASS:	32
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	56
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	66
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	107
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	110
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
MASS:	115
TIME in S./CYCLE :	1.0
H.V.VOLTAGE:	0.0
TIME PERIOD (Sec):	2400



SAMPLE: B 622-1

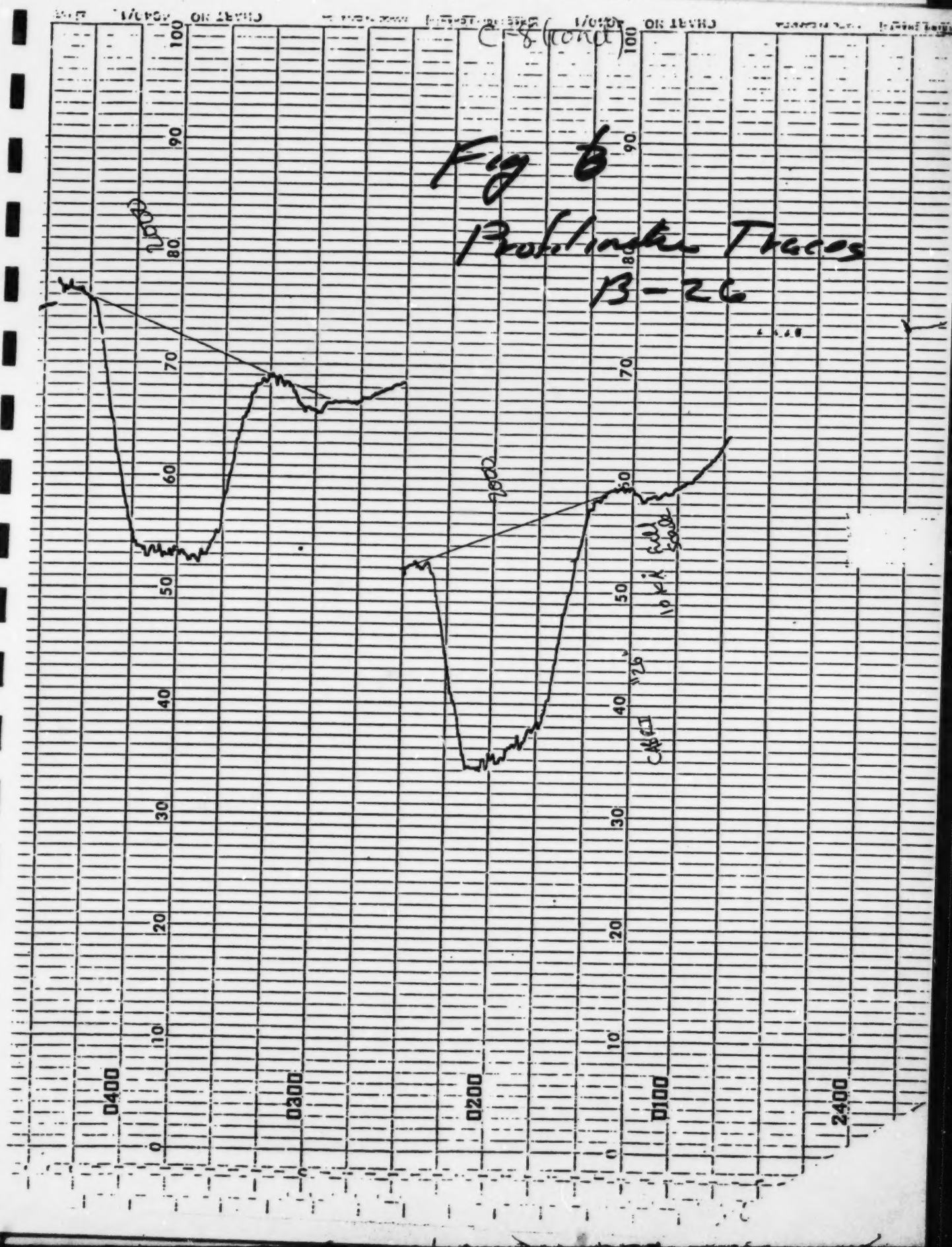


Fig 6
Probe Traces
B-26
Load
Load
B-26
10 Mil Full Scale
0400
0300
0200
0100
2400

Table 3 Summary of Sylolerit Stds. W-Ix

		Data	I Jan 86 A-1	II Feb 3 B-2	III Feb 3 A-1	IV Feb 8	I Feb 17	VI Feb 24	March	March 12	May 9	Mean values
30	Ag	490 ppm			2.60×10^{-2}	2.95×10^{-2}	3.21×10^{-2}	3.446×10^{-2}	2.81×10^{-2}	2.59×10^{-2}	2.24×10^{-2}	2.84×10^{-2}
	In	1140 ppm			4.50×10^{-1}	4.9×10^{-1}	6.01×10^{-1}	6.37×10^{-1}	4.77×10^{-1}	4.47×10^{-1}	5.76×10^{-1}	4.96×10^{-1}
31	Ag	550 ppm			1.29×10^{-2}	1.34×10^{-2}					9.53×10^{-3}	1.19×10^{-2}
	In	570			2.79×10^{-1}	2.97×10^{-1}					1.85×10^{-1}	2.37×10^{-1}
32	Ag	270 ppm			7.37×10^{-3}		9.46×10^{-3}	7.65×10^{-3}	7.36×10^{-3}	7.84×10^{-3}	6.54×10^{-3}	7.37×10^{-3}
	In	280			1.32×10^{-1}		1.05×10^{-1}	1.22×10^{-1}	1.23×10^{-1}	1.03×10^{-1}	1.02×10^{-1}	1.15×10^{-1}
33	Ag	110 ppm			3.70×10^{-3}	3.45×10^{-3}					2.65×10^{-3}	3.22×10^{-3}
	In	110			6.22×10^{-2}	4.20×10^{-2}					5.55×10^{-2}	4.32×10^{-2}
34	Ag	55 ppm			1.66×10^{-3}	1.94×10^{-3}	1.96×10^{-3}	2.27×10^{-3}	1.95×10^{-3}	3.78×10^{-3}	2.11×10^{-3}	2.09×10^{-3}
	In	57			2.58×10^{-2}	2.41×10^{-2}	2.00×10^{-2}	2.82×10^{-2}	2.41×10^{-2}	2.34×10^{-2}	2.17×10^{-2}	2.39×10^{-2}
35	Ag	16 ppm									9.02×10^{-4}	
	In	17									7.60×10^{-3}	
36	blank										7.29×10^{-4}	
											1.31×10^{-3}	

C-8 (cont'd)

Block B
Sphalerite
01
02
03
04
05
07A
07B
08
11
12
13
14
15
18
19
20
22
24
24A
25
25A
601
603
605
621
623
625

C-8 (cont'd)

Table 2 Summary of Analytical Results

Specimen	Silver (ug/g)	Indium ug/g
B 1-A	71 ± 2 + (63)	93 ± 1 (92)
B 3-A	297 ± 20 (325)	275 ± 11 (250)
B 4-A	59 ± 2 (228)	284 ± 6 (850)
B 4A-A	110 ± 2 (*)	674 ± 130 (*)
B 5-A	48 ± 1 (*)	17 ± 1 (*)
B 7-A	43 ± 2 (35)	54 ± 3 (43)
B 10-A	36 ± 2 (36)	802 ± 23 (800)
B 13-A	42 ± 1 (40)	2164 ± 80 (1950)
B 14-A	14 ± 1	158 ± 5
B 18-A	41 ± 2 (31)	8090 ± 600 (6000)
B 19-A	43 ± 1	5840 ± 400
B 20-A	57 ± 2	1840 ± 150 (second block ba
B 20-B	46 ± 2	1617 ± 60
B 22-A	51 ± 2	4450 ± 100
B 24-A	40 ± 2	16 ± 1

+ one standard deviation

* only one block analyzed

C-8(cont).

Table 2 Summary of Analytical Results

Specimen	Silver (ug/g)	Indium ug/g
B 1-A	71 ± 2 + (63)	93 ± 1 (92)
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B 7-A	43 ± 2 (35)	54 ± 3 (43)
B 10-A	36 ± 2 (36)	802 ± 23 (800)
B 13-A	42 ± 1 (40)	2164 ± 80 (1950)
B 14-A	14 ± 1	158 ± 5
B 18-A	41 ± 2 (31)	8090 ± 600 (6000)
B 19-A	43 ± 1	5840 ± 400
B 20-A	57 ± 2	1840 ± 150 (second block bad
B 20-B	46 ± 2	1617 ± 60
B 22-A	51 ± 2	4450 ± 100
B 24-A	40 ± 2	16 ± 1

\+ one standard deviation

* only one block analyzed

C-9

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

C-9

October 29, 1984

Dr. Louis J. Cabri
Canada Centre for Mineral & Energy Technology
Energy Mines and Resources
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis:

As requested in your letter of April 6 1984, we have analyzed zinc sulphide grains 6, 8 and 10 of Block A (25/10/83). As you suggested, the reference standards used came from those in block W-A.

For these runs, the O^- beam was adjusted to a diameter of 30um, as measured by its secondary ion image. The primary current used was normally ∿12na. The same profiling procedure was used as was reported in my letter of October 18/84.

The calibration plot obtained from the WA reference materials is shown in Figure 1. The data was treated by a least squares program, which was run both with and without the points from #607. The curvature in the fitted line arises from the use of log graph paper. If plotted on a linear scale the curvature would not be present. The accuracy at the 1 ug/g is ∿± 10 ug/g. It would obviously be of great benefit if a reliable standard in the range of 10 ug/g could be obtained, so that determinations in the range of 1-10 ug could be made with greater accuracy.

The raw data for the analysis of specimens 6, 8 and 10 are attached, and the ratios and concentrations are given in Tables I and II. Looking at the series of 10 ion intensity ratio results for each spot on specimen 6 (labelled BL-AZ-6), a gradual decrease in concentration is observed, which becomes more stable during the last 3-4 analyses. From point to point on specimen 6 the ratio varies by ± 100%. On specimens 8 and 10 higher silver/zinc ratios are observed as was earlier reported in my letter of January 24 1984.

In summary, then: it is clearly possible to distinguish differences in silver content near the1 ug/g level in sphalerites. The precision of the measurement appears to be in range of ± 100% and this likely results largely from local heterogeneties. The accuracy of the measurement is considerably poorer, and this stems from the unavailability of reference standards in the range of interest.

Best regards
yours very truly

N. S. Mc Intyre

c.c. D. Johnston

ing 603, 604, 606, 607

ppm = 38.96 * (107/66 * 10^3) - 62.56.

C-9

BL-A2-6-1
Point 1

CNTS 107/66 *1000	PPM AG
.0873	<1
.0812	<1
.0801	<1
.0733	<1
.0737	<1
.0680	<1
.0657	<1
.0632	<1
.0627	<1
.0580	<1

BL-A2-6-2
Point 2

CNTS 107/66 *1000	PPM AG
.1232	<1
.1197	<1
.1174	<1
.1059	<1
.1027	<1
.0936	<1
.1043	<1
.0866	<1
.0807	<1
.0871	<1

BL-A2-6-3
Point 3

CNTS 107/66 *1000	PPM AG
.0791	<1
.0704	<1
.0650	<1
.0637	<1
.0612	<1
.0563	<1
.0524	<1
.0525	<1
.0528	<1
.0516	<1

BL-A2-6-4
Point 4

CNTS 107/66 *1000	PPM AG
.0821	<1
.0696	<1
.0566	<1
.0527	<1
.0470	<1
.0413	<1
.0413	<1
.0356	<1
.0338	<1

.0323 <1

C-9

BL-A2-8-1

CNTS 107/66	*1000	PPM AG
.2164		<1
.1936		<1
.1765		<1
.1612		<1
.1515		<1
.1466		<1
.1465		<1
.1514		<1
.1460		<1
.1505		<1

BL-A2-8-2

CNTS 107/66	*1000	PPM AG
.1892		<1
.1690		<1
.1530		<1
.1539		<1
.1496		<1
.1435		<1
.1380		<1
.1470		<1
.1400		<1
.1357		<1

BL-A2-10-1

CNTS 107/66	*1000	PPM AG
.3782		<1
.3426		<1
.4058		<1
.4371		<1
.4143		<1
.4776		<1
.5026		<1
.4442		<1
.4927		<1
.4691		<1

BL-A2-10-2

CNTS 107/66	*1000	PPM AG
.6869		<1
.8242		<1
.8171		<1
.7957		<1
.7043		<1
.6232		<1
.6458		<1

Table II C-9 (cont'd)

Using 603, 604, 606

ppm Ag = $37.13 * (107/66 * 10^3) - 17.83$

ppm In = $16.12 * (115/66 * 10^2) + 53.64$

B1-A2-6-1

CNTS 107/66 *1000	PPM AG
.0873	<1
.0812	<1
.0801	<1
.0733	<1
.0737	<1
.0680	<1
.0657	<1
.0632	<1
.0627	<1
.0580	<1

B1-A2-6-2

CNTS 107/66 *1000	PPM AG
.1232	<1
.1197	<1
.1174	<1
.1059	<1
.1027	<1
.0936	<1
.1043	<1
.0866	<1
.0807	<1
.0871	<1

B1-A2-6-3

CNTS 107/66 *1000	PPM AG
.0791	<1
.0704	<1
.0650	<1
.0637	<1
.0612	<1
.0563	<1
.0524	<1

C-91 (cond)

.0525	<1
.0528	<1
.0516	<1

B1-A2-6-4

CNTS 107/66 *1000	PPM AG
.0821	<1
.0696	<1
.0566	<1
.0527	<1
.0470	<1
.0413	<1
.0413	<1
.0356	<1
.0338	<1
.0323	<1

B1-A2-8-1

CNTS 107/66 *1000	PPM AG
.2164	<1
.1936	<1
.1765	<1
.1612	<1
.1515	<1
.1466	<1
.1465	<1
.1514	<1
.1460	<1
.1505	<1

B1-A2-8-2

CNTS 107/66 *1000	PPM AG
.1892	<1
.1690	<1
.1530	<1
.1539	<1
.1496	<1
.1435	<1
.1380	<1
.1470	<1
.1400	<1
.1357	<1

B1-A2-10-1

CNTS 107/66 *1000	PPM AG
.3782	<1
.3426	<1
.4058	<1
.4371	<1
.4143	<1

C-9 (cond)

.4776	<1
.5026	<1
.4442	<1
.4927	<1
.4691	<1

B1-A2-10-2

CNTS 107/66	*1000	PPM AG
.6869		7
.8242		12
.8171		12
.7957		11
.7043		8
.6232		5
.6458		6
.6426		6
.6498		6
.6653		6

C-9 (cond)

B1-A2-6-1

CNTS 115/66	*100	PPM AG
.0623		54
.0622		54
.0646		54
.0584		54
.0646		54
.0613		54
.0651		54
.0615		54
.0675		54
.0640		54

B1-A2-6-2

CNTS 115/66	*100	PPM AG
.0755		54
.0814		54
.0819		54
.0816		54
.0819		54
.0803		54
.0859		55
.0797		54
.0813		54
.0811		54

1.0000 A2-6-3

CNTS 115/66	*100	PPM AG
.0760		54
.0770		54
.0800		54
.0771		54
.0796		54
.0766		54
.0765		54
.0731		54
.0746		54
.0793		54

B1-A2-6-4

CNTS 115/66	*100	PPM AG
.0711		54
.0725		54
.0687		54
.0707		54
.0700		54
.0714		54
.0695		54
.0661		54

.0697	54

C-9 (cont'd)

B1-A2-8-1

CNTS 115/66 *100	PPM AG
.0729	54
.0763	54
.0752	54
.0749	54
.0726	54
.0714	54
.0694	54
.0706	54
.0682	54
.0654	54

B1-A2-8-2

CNTS 115/66 *100	PPM AG
.0571	54
.0557	54
.0554	54
.0562	54
.0547	54
.0543	54
.0541	54
.0548	54
.0544	54
.0556	54

B1-A2-10-1

CNTS 115/66 *100	PPM AG
.0648	54
.0645	54
.0628	54
.0622	54
.0644	54
.0647	54
.0701	54
.0665	54
.0654	54
.0678	54

B1-A2-10-2

CNTS 115/66 *100	PPM AG
.0779	54
.0847	55
.0871	55
.0870	55
.0844	55
.0825	54
.0824	54

.0817	54
.0807	54
.0767	54

C-9 (cond)

Oct 9/84 Sphalerite W-A

std #	ppm Ag	$107/66 * 10^3$	ppm In	$115/66 * 10^2$
603	1090	29.74	1140	67.33
604	550	15.56	570	32.26
605	~~270~~	.2065	blank	.07285
606	270	7.581	280	13.90
607-1	110	5.395	110	12.32
2	110	7.214	110	17.04
3	110	6.205	110	14.28

Least squares analysis

① Points 603, 604, 606, 607

Ag m = 38.96 b = -62.56

In m = 17.50 b = -25.52

② Points 603, 604, 606

Ag m = 37.13 b = -17.83

In m = 16.12 b = 53.64

$$ppm = 37.13 \times 10^{-6} \frac{107}{66} \times ND - 17.8 \quad |ppm =$$

$$\frac{1 + 17.8}{37.13} \qquad 20$$

$$\frac{100 + 17.8}{37.13}$$

1000

ERROR 17 IN LINE 4035 C-9(cond)

MEASUREMENT CONDITIONS

SAMPLE: B1.A-603-1

MASS:	66	31786	6
MASS:	107	40904	5
MASS:	115	42447	5

REFERENCE MASS: 66
MEASUREMENT TIME: 10
WAITING TIME : 0
NUMBER OF CYCLES: 5
NUMBER OF BLOCKS: 2
CALIBRATION: Manual

RESULTS

BLOCK No: 1

CYCLE No: 1

66/REF.-->	+1.000E+00	CNTS-->1.5116E+07
107/REF.-->	+2.865E-02	CNTS-->4.3300E+05
115/REF.-->	+6.264E-01	CNTS-->9.4685E+06
		BKGD-->3.000E+00

CYCLE No: 2

66/REF.-->	+1.000E+00	CNTS-->1.4669E+07
107/REF.-->	+2.899E-02	CNTS-->4.2528E+05
115/REF.-->	+6.500E-01	CNTS-->9.5343E+06
		BKGD-->1.000E+00

CYCLE No: 3

66/REF.-->	+1.000E+00	CNTS-->1.4257E+07
107/REF.-->	+2.930E-02	CNTS-->4.1773E+05
115/REF.-->	+6.537E-01	CNTS-->9.3195E+06
		BKGD-->0.000E+00

CYCLE No: 4

66/REF.-->	+1.000E+00	CNTS-->1.3673E+07
107/REF.-->	+2.938E-02	CNTS-->4.0167E+05
115/REF.-->	+6.562E-01	CNTS-->8.9721E+06
		BKGD-->0.000E+00

CYCLE No: 5

66/REF.-->	+1.000E+00	CNTS-->1.3448E+07
107/REF.-->	+2.934E-02	CNTS-->3.9452E+05
115/REF.-->	+6.599E-01	CNTS-->8.8750E+06
		BKGD-->0.000E+00

MEAN RATIO

REFERENCE PEAK MASS: 66

MASS: 66	MEAN:	+1.000E+00	STD DEV : +0.000E+00
MASS: 107	MEAN:	+2.913E-02	STD DEV : +3.104E-04

MEAN RATIO

REFERENCE PEAK MASS: 66 C-9 (corrected)

MASS: 66	MEAN:	+1.000E+00	STD DEV :	+0.000E+00
MASS:107	MEAN:	+2.913E-02	STD DEV :	+3.104E-04
MASS:115	MEAN:	+6.492E-01	STD DEV :	+1.327E-02

ATOMIC o/o

MASS: 66	PER CENT	: +59.582
MASS:107	PER CENT	: +1.736
MASS:115	PER CENT	: +38.683

WEIGHT (o/o)

MASS: 66	PER CENT	: +45.904
MASS:107	PER CENT	: +2.168
MASS:115	PER CENT	: +51.928

****

BLOCK No: 2

****

CYCLE No: 1

66/REF.-->	+1.000E+00	CNTS-->1.3328E+07
107/REF.-->	+2.947E-02	CNTS-->3.9279E+05
115/REF.-->	+6.677E-01	CNTS-->8.8990E+06
		BKGD-->0.000E+00

CYCLE No: 2

66/REF.-->	+1.000E+00	CNTS-->1.3387E+07
107/REF.-->	+2.972E-02	CNTS-->3.9790E+05
115/REF.-->	+6.642E-01	CNTS-->8.8921E+06
		BKGD-->1.000E+00

CYCLE No: 3

66/REF.-->	+1.000E+00	CNTS-->1.2981E+07
107/REF.-->	+2.998E-02	CNTS-->3.8915E+05
115/REF.-->	+6.835E-01	CNTS-->8.8718E+06
		BKGD-->1.000E+00

CYCLE No: 4

66/REF.-->	+1.000E+00	CNTS-->1.3128E+07
107/REF.-->	+2.961E-02	CNTS-->3.8869E+05
115/REF.-->	+6.663E-01	CNTS-->8.7472E+06
		BKGD-->1.000E+00

CYCLE No: 5

66/REF.-->	+1.000E+00	CNTS-->1.2795E+07
107/REF.-->	+2.993E-02	CNTS-->3.8298E+05
115/REF.-->	+6.849E-01	CNTS-->8.7628E+06
		BKGD-->1.000E+00

MEAN RATIO

REFERENCE PEAK MASS: 66

MASS: 66	MEAN:	+1.000E+00	STD DEV :	+0.000E+00
MASS:107	MEAN:	+2.974E-02	STD DEV :	+2.145E-04
MASS:115	MEAN:	+6.733E-01	STD DEV :	+9.994E-03

ATOMIC o/o

MASS: 66	MEAN:	+1.000E+00	STD DEV : +0.000E+00
MASS:107	MEAN:	+2.974E-02	STD DEV : +2.145E-04
MASS:115	MEAN:	+6.733E-01	STD DEV : +9.994E-03

ATOMIC o/o

MASS: 66	PER CENT :	+58.718
MASS:107	PER CENT :	+1.746
MASS:115	PER CENT :	+39.536

C-4 (cont'd).

WEIGHT (o/o)

MASS: 66	PER CENT :	+45.016
MASS:107	PER CENT :	+2.171
MASS:115	PER CENT :	+52.813

BLOCKS AVERAGE

66/Ref--->	AVERAGE	+1.000E+00	STD DEV	+0.000E+00
107/Ref--->	AVERAGE	+2.944E-02	STD DEV	+4.333E-04
115/Ref--->	AVERAGE	+6.613E-01	STD DEV	+1.703E-02

SAMPLE:B1.A-603-1
DATE:9/10/84
PRIMARY IONS : OXYGEN -
SECONDARY IONS : +
DEAD TIME: 129.30E-09 sec.

PRIMARY CURRENT : 12 ~~20~~ NANO AMP
HIGH VOLTAGE : 11.7 KILOVOLTS
RASTERING : 0 MICRONS
TRANSFER OPTICS : 150 MICRONS
FIELD APERTURE : (1) 2 3
CONTRAST APERTURE : 4 (3) 2 1

0

C-10

Energy, Mines and Resources Canada
Research and Technology
Canada Centre for Mineral and Energy Technology,
555 Booth Street,
Ottawa, Canada
K1A 0G1

Énergie, Mines et Ressources Canada
Recherche et Technologie
Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa, Canada
K1A 0G1

Your file Votre référence

Our file Notre référence

BY COURIER

November 28, 1984

Dr. N.S. McIntyre
Technical Director
Surface Science
Western Centre for Interdisciplinary
Studies in Chemical Physics
University of Western Ontario
London, Ontario
N6A 3K7

Dear Stewart:

I have been studying some of the SIMS analyses of sphalerites to try to assess the reproducibility and accuracy of the results. I have plotted the results for Ag on the standards from Jan. 26 to July 10, 1984 and have drawn a few eye-balled curves as you will see on the enclosed copy. The spread of values within Block W-A standards is disturbing. Also note the lack of correspondence between two July 10 analyses (Block B) to any of the earlier analyses done on Block W-A. I wonder if the fact that standard 598 has no Fe may partly account for that? On the other hand, it does not explain the spread for the standards within Block W-A.

I have also made a table comparing the results from SIMS, micro-PIXE and electron microprobe for the exact same grains and exactly same spots on the grains. The sequence was micro-PIXE followed by electron probe and finally by SIMS. Unfortunately the comparison between the three techniques is only possible for In (for 5 grains) but I believe there is a cause for concern as the only fairly good correspondence occurs between micro-PIXE and electron probe. If we look at the rest of the results, SIMS only sometimes corresponds fairly well with micro-PIXE (4 out of 14 for Ag, 2 out of 14 for In). Since there are no apparent trends, I cannot interpret these few apparently comparable results.

I shall look forward to your comments and suggestions. I have two sphalerite sections ready for you if we can come up with ideas on how the apparent variations and lack of agreement can be improved.

Best wishes,

Louis J. Cabri

Encl.
LJC/tj

Canada

C-10(cont'd)

No.	Indium			Silver	
	SIMS	PIXE	Electron Probe	SIMS	PIXE
B1-A	93	n.d.	-	71	n.d.
B3-A	276	87	-	297	49
B4-Ax	284	31	-	59	287
B4A-Ay	674	35	-	110	329
B5-A	18	n.d.	-	48	28
B7-A	55	n.d.	-	43	n.d.
B10-A	804	121	-	37	25
B13-A	2164	618	1000	42	45
B14-A	158	60 61	-	14	n.d.
B18-A	8090	2035 2083	2400	41	n.d.
B19-A	5843	1636 1638 1638	2100	43	72
B20-A	1843	362	700	58	36
B22-A	4452	955	1200	51	111
B24-A	17	n.d.	-	40	n.d.
		n.d.=<16	To be changed		n.d.=<12

C-10

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

Research and Technology
Recherche et Technologie

Canada Centre for Mineral and Energy Technology,
555 Booth Street,
Ottawa, Canada
K1A 0G1

Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa, Canada
K1A 0G1

Your file Votre référence

Our file Notre référence

December 11, 1984

BY COURIER

C-10

Dr. N.S. McIntyre
Technical Director
Surface Science
Western Centre for Interdisciplinary
Studies in Chemical Physics
University of Western Ontario
London, Ontario
N6A 3K7

Dear Stewart:

Further to our brief discussions in my office on the 7th concerning the sections W-A sp (Dec. 84) and Block B sphalerite that you took with you.

1. W-A sp (Dec. 84)
This section has been repolished and contains an additional standard (No. 39). The attached sheet gives a new listing of all standards for easy reference. The new photograph with circled areas for analysis is also enclosed. You can analyse circled areas in any (or all) unknowns except for Nos. 1-4 and 8-9.

2. Block B sphalerite
This section has not been modified from when you had it last. A photocopy of the photograph is enclosed and you could analyse most samples in circled areas which have not been previously analysed. However, please don't bother with No. 26. I couldn't find the original photo so hope you still have it.

3. We agreed that you would first analyse the full set of standards in W-A (Nos. 30-36 inclusive plus 39) in order to establish a new calibration curve but using Fe + Zn. You could then analyse the standards in B and see how they compare. We also agreed that you would ensure that analytical conditions remained the same for all analyses. For the unknowns, you would do Fe+Zn too, and maybe Cd as well.

.../2

Canada

4. I am returning printouts from previous analyses labelled "WA- sphalerite later Res.", "ZnS-specimen Isolated" and "Standard Sets ZnS".

I shall look forward to the new results. With best wishes for Christmas and for the New Year.

Yours sincerely,

Louis J. Cabri

Encl.
LJC/tj

P.S. I shall look forward to a few reprints of our paper.

C-10 (cond)

December 11, 1984

Standards

Section W-A Sp (Dec. 84)

No.	(Old No.)	Zn	wt.% Fe	Ag	In
30	(603)	64.20	2.56	0.109	0.114
31	(604)	64.32	2.54	0.055	0.057
32	(606)	64.37	2.54	0.027	0.028
33	(607)	64.38	2.55	0.011	0.011
34	(608)	64.39	2.56	0.0055	0.0057
35	(609)	64.41	2.55	0.0016	0.0017
36	(605)	64.19	2.75	-	-
39	(sp20)	62.07	4.75	-	-

Section B

No.	Zn	wt.% Fe	Ag	In
598	66.90	-	0.114	0.119
601	67.10	-	-	-
603	64.20	2.56	0.109	0.114
605	64.19	2.75	-	-
622	63.60	3.19	0.073	0.079
621	63.75	3.17	-	-

Louis Cabri 85

C-11

Surface Science

The Surface Science Laboratory, The University of Western Ontario.
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

C-11

1985/01/11

Dr. Louis J. Cabri
Canada Centre for Mineral & Energy Technology
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis,

In response to your letter of December 11th we enclose results for the analytical response curves for Ag and In in block W-A and W-B standards. The results were obtained under the following instrumental conditions:

Field Aperture - 1
Contrast Aperture - 3
Primary Current - 20 nA
Primary spot size - 40-50 um
Non rastered O primary beam

The results for both offset conditions are included as Set 1 and Set 2. Set 1 was collected with no offset voltage, while Set 2 had an offset of 75 volts. You will note that we now collect the data in depth profile form. In my opinion this has enormous advantages over the earlier "Isotope" format, since the evaluation of each ion intensity can be monitored so much easier. The measurement statistics are unaffected by the change in program.

As a result of using the new settings, it is clear from the data in Set 1, that the slowly altering Ag/Zn intensity ratio which plagued us previously, is now much less of a problem. The depth profiles in Set 1 show that the relative intensites of Ag, In & Zn remain relatively constant and it is thus possible to take a ratio measurement with confidence not long after the profile has begun. It should be noted that the WA standards used had already been presputtered in a preliminary run, and that for these earliest results, a very thin surface-modified layer was detected. This layer was penetrated in ~2 minutes. For the results demonstrated in Set 1, virtually no surface modified layer was detected. For the Set 1 results, the intensity ratios of $^{107}Ag/66_{Zn}$ and $^{115}In/66_{Zn}$ were determined by the program and are printed out for each cycle at the end of each data sheet. For present purposes, an average of the last five cycles was used.

C-11 (cont'd)

The averages of these results are plotted in Figure 1 against the given concentrations for standards 30-35. Because of some confusion we did not run 36 and 39. The correlation shown for all standards is quite good.

A second set of results (Set 2) was subsequently obtained using an offset voltage of 75 volts. During this run, ^{54}Fe and ^{67}Zn were also measured. The Set 2 plots for $^{107}Ag/^{66}Zn$ and $^{115}In/^{66}Zn$ are shown as red dots on Figure 1. The correlation is reasonable for all standards except for #34.

Following your instructions the standards set in Block B were then analyzed under the same conditions as in Set 1. The data for these standards: 622 (2 runs), 598 (2 runs), and 603, is included as Set 3. It appears that the curve for points 622 and 603 (standards with iron) lies off the curve for Set 2. Further, it appears that the data points for 598 (low iron standard) may be displaced from this curve, but the statistics are poor. Both complete sets of standards should be run again very carefully taking several independent measurements on each one.

The Set 2 results were also plotted for $^{107}Ag/^{54}Fe$ and $^{107}Ag/^{67}Zn$. The latter ratio was done to determine if the strong ^{66}Zn signal is sometimes susceptible to dead time losses. The plots for $^{107}Ag/^{54}Fe$ (and $^{115}In/^{54}Fe$) show that good correlation (data not taken for point 30) and that the discrepancy for point 34 noted above is not present - presumably because the measurement problem lay with the zinc intensity. The correlation for the $^{107}Ag/^{67}Zn$ is not as good. Thus it appears that ^{54}Fe is at least as reliable a reference in these standards as zinc. Perhaps on occasions it is even better. In our preliminary depth profiles we found several occasions where the silver intensity followed the iron intensity more closely than it did the zinc. I recommend that in all future work with sphalerites we have two separate calibration curves and calculate the result from each one.

As I indicated above we spent some time in December in a preliminary round, developing software and testing settings. As part of this we ran some trial depth profiles of B-3 and B-4 - two of the specimens whose SIMS anlayses differed sharply with the PIXE data. Figures 3 and 4 show the profiles for silver, indium, iron, zinc and cadmium. The silver concentration varies widely in these specimens and this may go some distance toward explaining the difference in the silver concentrations detected by SIMS and PIXE. SIMS would determine the concentration at a local point, while PIXE represents more of an average. However in the specimen investigated there was no similar variation in indium concentration, while from your results it appears that there is a discrepancy between techniques for indium as well.

We propose to do the following experiments next:

(1) Careful determination of both standard curves (A¢B) again, plotting Ag and In using both Zn-66 and Fe-54.

(2) Analysis of the B series of unknowns using both Fe and Zn as references. Measurement of the crater depths to determine the microdistribution.

I will be in Ottawa next week from the 16th to the 18th at Jim Brown's lab at Bells Corners. Call me with your comments. (996-4570)

With best wishes for '85.
Yours very truly,

N.S. McIntyre
Director
Surface Science Western

G-125 LOGARITHMIC 5 X 3 CYCLES
SPECIFY TRACING OR DRAWING PAPER

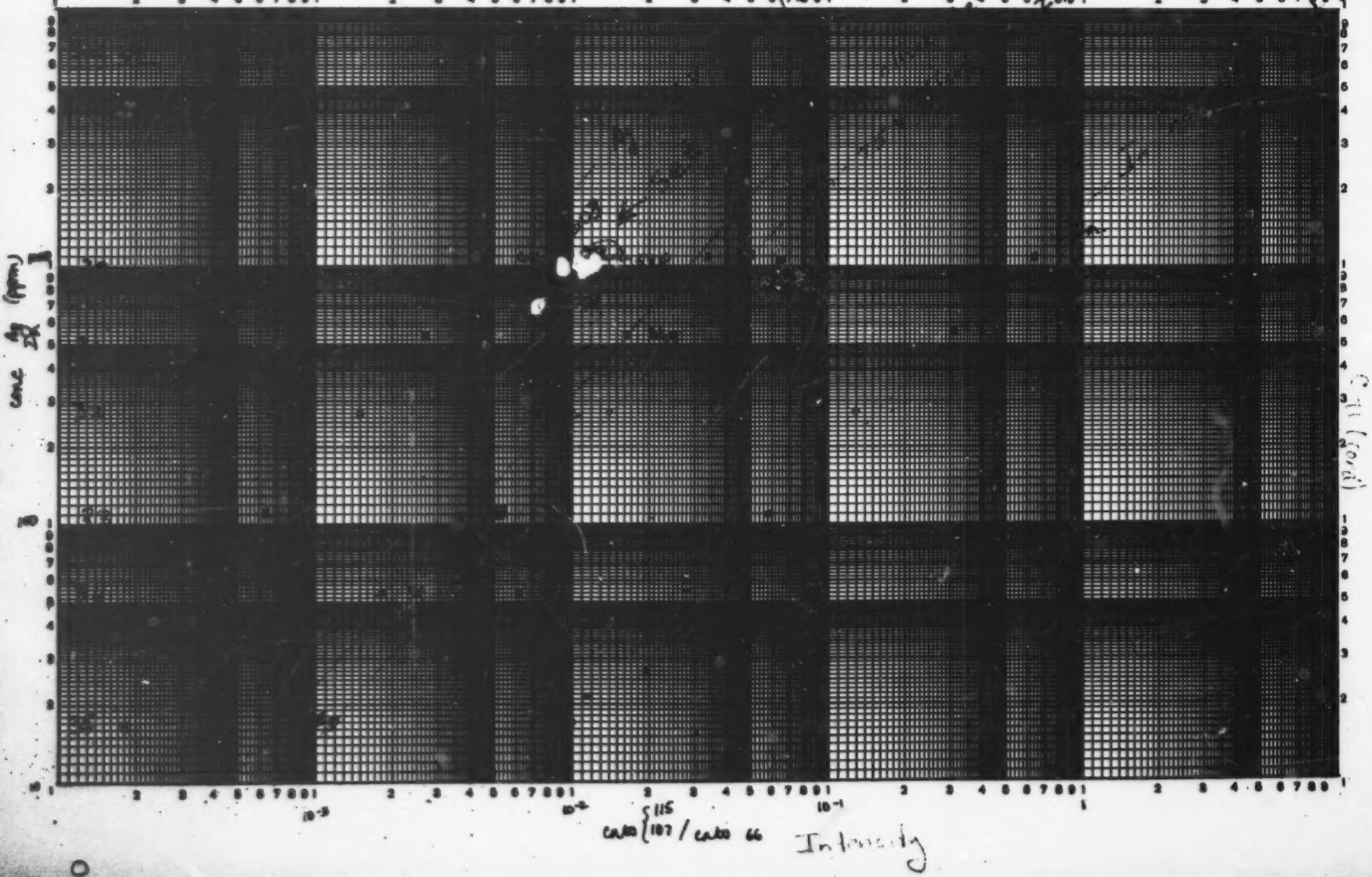
GRAPHIC CONTROLS CANADA LTD.
MADE IN CANADA

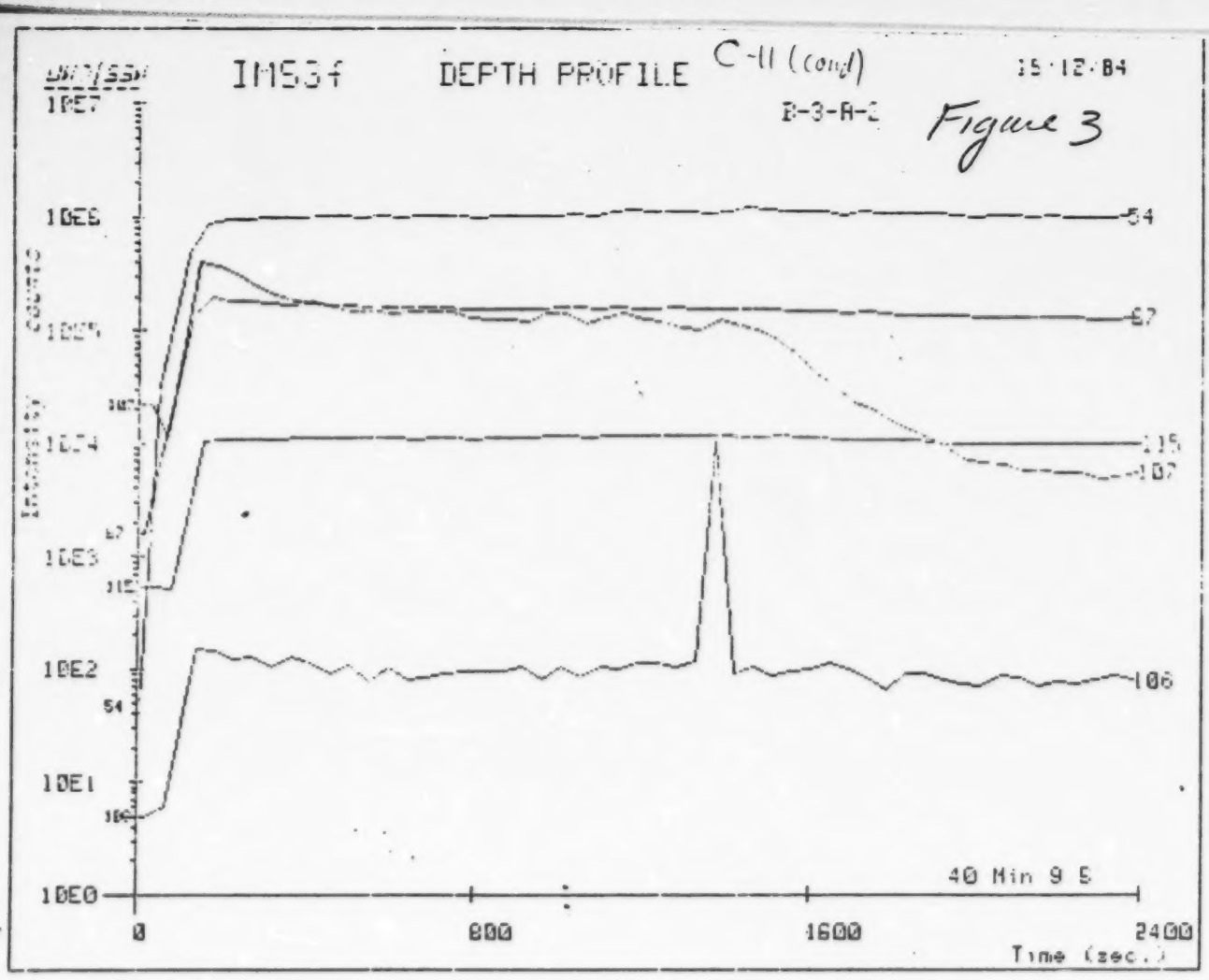
Figure 1

WA 81185 20nA FA 1 cA-3

Ref mass = 66

O - Black B Sand





SAMPLE:B-3-A-2
DATE:19/12/84
PRIMARY IONS : OXYGEN -
SECONDARY IONS : +
DEAD TIME: 500.00E-10 sec.

PRIMARY CURRENT : 41 NANO AMP
HIGH VOLTAGE : 11.6 KILOVOLTS
RASTERING : 6 MICRONS
TRANSFER OPTICS : 25 MICRONS
FIELD APERTURE : 1 2 3
CONTRAST APERTURE : 4 3 2 1

Manual

MASS NUMBER : 54.00 COUNT TIME : 5.00 OFFSET VOLTAGE : 0.00
M.F.P.: 28608 2

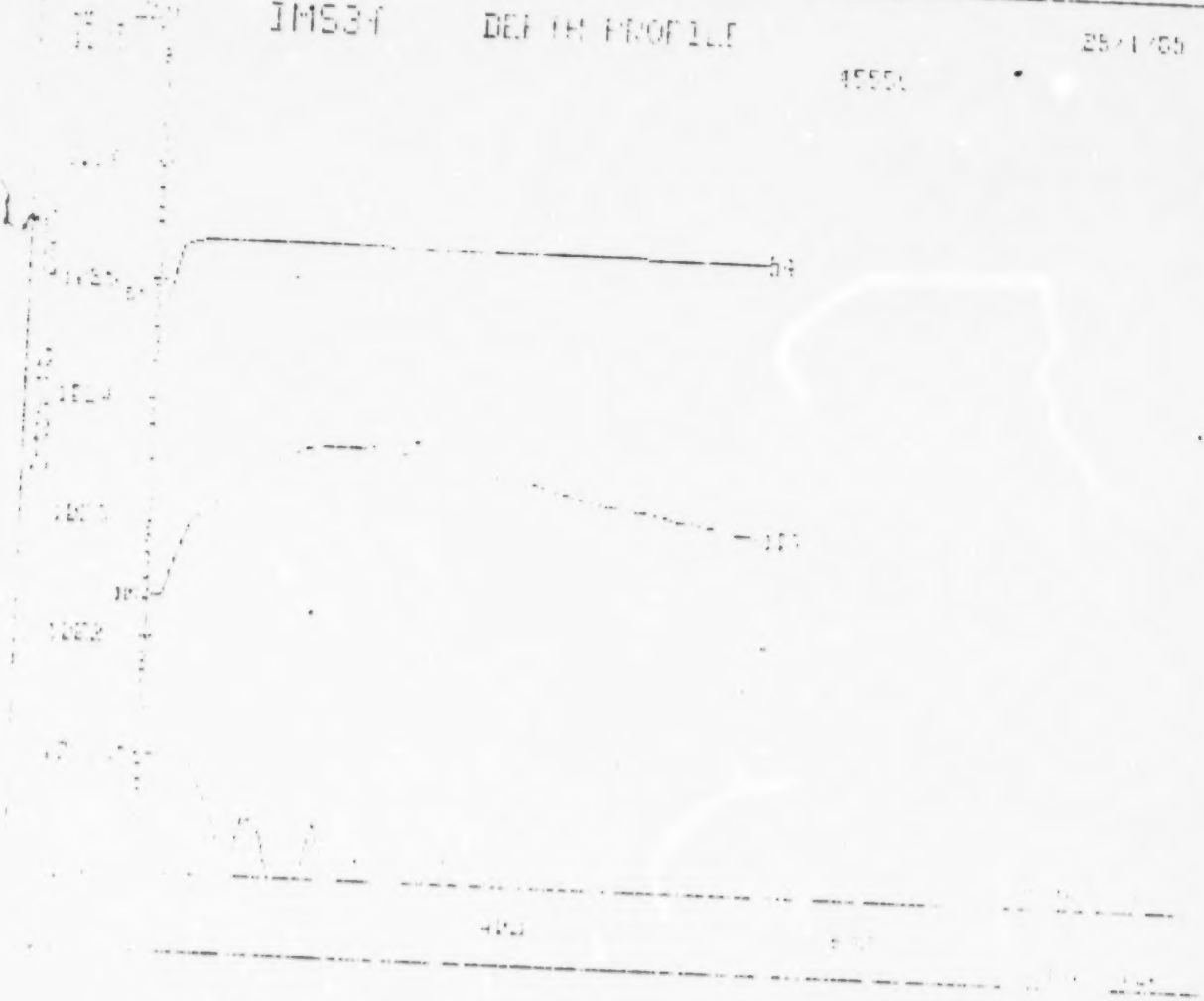
TIME(SEC)	COUNT	TIME(SEC)	COUNT	TIME(SEC)	COUNT	TIME(SEC)	COUNT
7	47	50	31650	126	485155	172	877109
210	991492	264	947865	310	970911	356	1011932
401	1008442	447	1017169	493	1022276	539	1012351
585	1015105	630	983612	676	1046169	722	1018449
767	1011478	813	980280	859	1050276	905	1016571

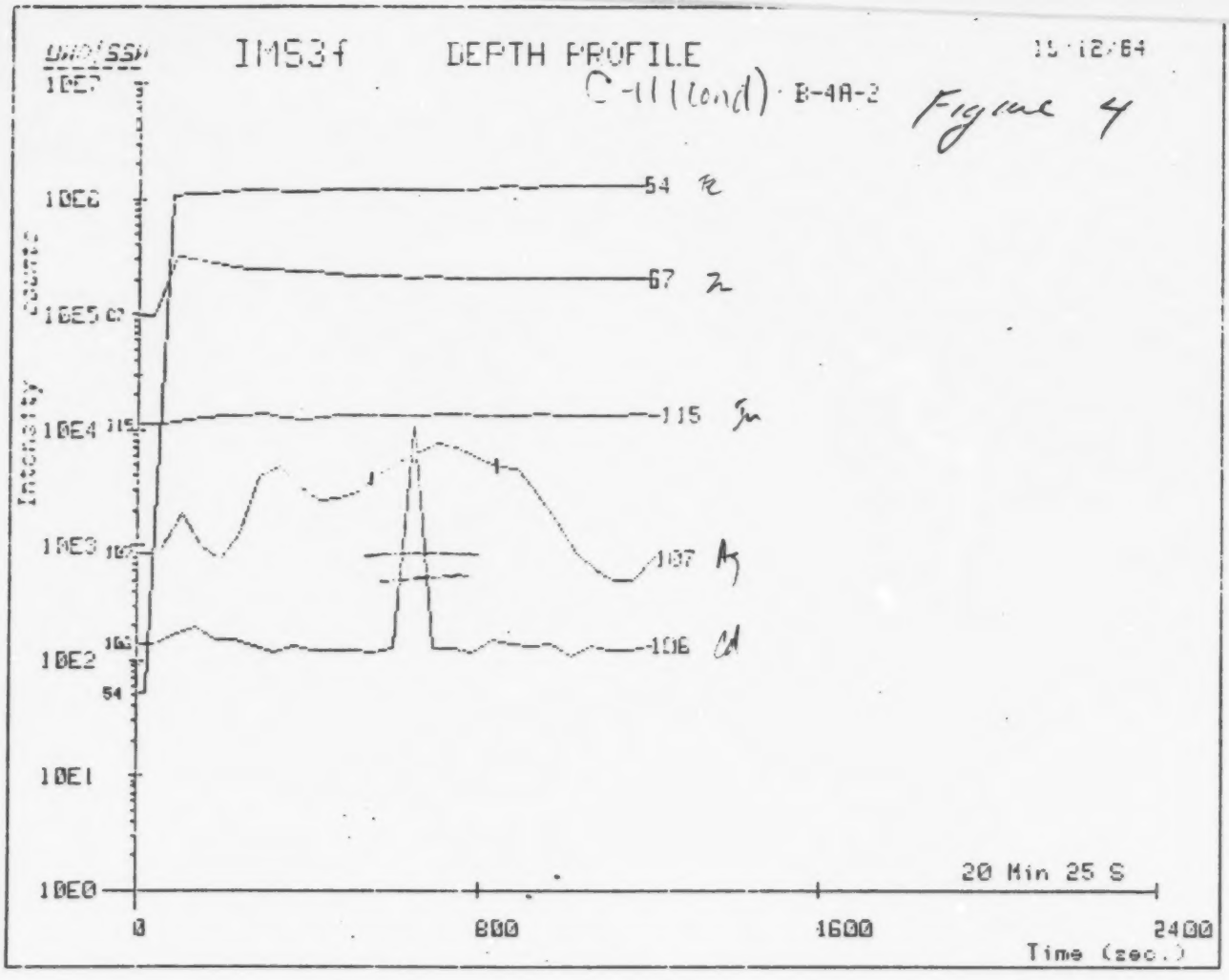
C-11 (cont'd)

.00E-10	
MASS:	54
TIME in S. CYCLE :	.5
H.V.VOLTAGE:	-100.0
MASS:	107
TIME in S./CYCLE :	3.0
H.V.VOLTAGE:	-100.0
MASS:	109
TIME in S./CYCLE :	3.0
H.V.VOLTAGE:	-100.0
TIME FERIOD (Sec):	120m

Implant spot mode

IMS3f DEPTH PROFILE 25/1/85





SAMPLE:B-4A-2
DATE:19/12/84
PRIMARY IONS : OXYGEN -
SECONDARY IONS : +
DEAD TIME: 500.00E-10 sec:

PRIMARY CURRENT : 41 NANO AMP
HIGH VOLTAGE : 11.6 KILOVOLTS
RASTERING : 0 MICRONS
TRANSFER OPTICS : 25 MICRONS
FIELD APERTURE : 1 2 3
CONTRAST APERTURE : 4 3 2 1

Manual

MASS NUMBER : 54.00 COUNT TIME : 5.00 OFFSET VOLTAGE : 0.00
M.F.P.: 28609 2

TIME(SEC)	COUNT	TIME(SEC)	COUNT	TIME(SEC)	COUNT	TIME(SEC)	COUNT
7	47	50	32650	126	-85:55	172	977105
218	951482	264	947865	310	970911	356	1011512
401	1008442	447	1027169	493	1032276	539	1023e1
585	1015105	630	983612	676	1046169	722	1028449
767	1018278	813	980280	859	1050276	905	1026571

18/12/84

C-11 (cont'd)

WA-30-A-1

	107/54*100	107/66*100
	1.8242	.3378
	1.7817	.3307
	1.8174	.3419
	1.7875	.3339
	1.8430	.3537
MEAN	1.8108	.3396
SD	.0257	.0089

WA-30-C-1

	107/54*100	107/67*100
	1.7397	2.2063
	1.6424	2.1545
	1.7242	2.1565
	1.7342	2.1790
	1.7100	2.2037
MEAN	1.7101	2.1800
SD	.0394	.0247

WA-31-A-1

	107/54*100	107/67*100
	3.3766	4.6795
	3.5398	4.8299
	3.5169	4.6985
	3.6809	5.0699
	3.9665	5.0445
MEAN	3.6162	4.8644
SD	.2236	.1854

WA-32-A-1

107/54*100	107/67*100
.9235	1.7294
1.0141	1.9218
.9512	1.8238
.9333	1.9336
1.0080	1.9997

C-11 (cont'd)

MEAN	.9660	1.8816
SD	.0423	.1058

WA-33-A-1

107/54*100	107/67*100
1.9123	3.5208
1.8672	3.4714
1.9913	3.8657
2.0130	3.8246
1.9442	3.8849
MEAN 1.9456	3.7135
SD .0589	.2004

WA-33-A-2

107/54*100	107/67*100
2.2658	7.0098
1.4970	7.0073
2.3757	7.5193
2.3900	7.5371
2.3514	7.4983
MEAN 2.1760	7.3144
SD .3826	.2795

WA-34-A-1

107/54*100	107/67*100
.0663	.2215
.0564	.2191
.0658	.2496
.0579	.2349
.0589	.2265
MEAN .0611	.2303
SD .0046	.0123

WA-35-A-1

C-11 (cont)

	107/54*100	107/67*100
	.0017	.0097
	.0022	.0124
	.0030	.0167
	.0006	.0040
	.0017	.0105
MEAN	.0018	.0107
SD	.0008	.0046

WA-36-A-1

	107/54*100	107/67*100
	20.4626	.0714
	29.3723	.0816
	48.3146	.0812
	66.1538	.0842
	77.1992	.0930
MEAN	48.3005	.0823
SD	23.9118	.0077

WA-39-A-1

	107/54*100	107/67*100
	.0535	.0792
	.0512	.0766
	.0505	.0789
	.0598	.0961
	.0582	.0949
MEAN	.0547	.0851
SD	.0041	.0095

B-26-A-1

	107/54*100	107/67*100
	1.7261	8.5027
	1.7390	8.8528
	1.5561	8.5732
	1.5858	8.4921
	1.6895	9.2314
MEAN	1.6593	8.7304
SD	.0833	.3159

C-11 (cont'd)

B-26-B-1

	107/54*100	107/67*100
	.9682	2.7039
	.8663	2.6892
	.8746	2.6902
	.9206	3.0132
	1.0015	3.1624
MEAN	.9262	2.8518
SD	.0585	.2218

B-598-1

	107/54*100	107/67*100
	.* 4013.99	8.9731
	.* 3773.15	8.6351
	.* 4142.16	9.8206
	.* 4486.14	9.1930
	.* 4259.81	9.3072
MEAN	***.**** 4163.23	9.1858
SD	264.9890	.4376

B-603-1

	107/54*100	107/67*100
	3.7864	6.2949
	4.3087	7.5686
	4.1906	7.0957
	4.5316	8.0758
	4.3720	8.3154
MEAN	4.2378	7.4701
SD	.2807	.8081

B-603-2

	107/54*100	107/67*100
	4.7300	7.5287
	4.5323	7.4957
	4.5774	7.9486
	4.7022	8.3869
	4.7366	8.5525

C-11 (cond)

MEAN	4.6557	7.9825
SD	.0943	.4828

B-605-1

107/54*100	107/67*100
.0158	.0316
.0145	.0302
.0132	.0287
.0158	.0358
.0138	.0324
MEAN .0146	.0317
SD .0011	.0026

B-621-1

107/54*100	107/67*100
.0388	.0667
.0369	.0634
.0396	.0693
.0395	.0707
.0377	.0699
MEAN .0385	.0680
SD .0011	.0029

B-622-1

107/54*100	107/67*100
2.1528	3.9479
2.2488	3.9283
2.1697	3.8815
2.2674	4.2167
2.2785	4.4414
MEAN 2.2235	4.0832
SD .0580	.2393

Energy, Mines and Resources Canada
Énergie, Mines et Ressources Canada

Research and Technology
Recherche et Technologie

Canada Centre for Mineral and Energy Technology,
555 Booth Street,
Ottawa, Canada
K1A 0G1

Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa, Canada
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C-12

Your file / Votre référence

Our file / Notre référence

BY COURIER

C-12

January 29, 1985

Dr. N.S. McIntyre
Technical Director
Surface Science
Western Centre for Interdisciplinary
Studies in Chemical Physics
University of Western Ontario
London, Ontario
N6A 3K7

Dear Stewart:

Thank you for your prompt phone reply to yesterday's letter. Enclosed are the data sheets you requested. I apologize for a mistake in my point 1 of yesterday's letter. The transfer optics were not different in set 3, only the primary current: i.e. 4-5 nanoamps versus 20 for sets 1 & 2.

Best wishes.

Yours sincerely,

Louis J. Cabri

Encl.
LJC/tj

P.S. I hope you will be able to book sufficient machine time soon so that the remaining contract money does not lapse as I won't be able to transfer it to next FY. Thanks.

Canada

C-13

Surface Science Western

The Surface Science Laboratory, The University of W... tern Ontario
Natural Sciences Centre, London, O... o N6A 5B
9) 679-2233

C-13

January 30, 1985

Dr. Louis J. Cabri
Research and Technology
Canada Centre for Mineral and Energy Technology
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis:

In response to your letter of January 28th I have the following replies:

(1) The same settings were used for all data taken, except for specimens B-3 and B-4. These latter specimens were run during preliminary trials of the new tests and were shown for qualitative purposes only.

(2) We can certainly try to plot a series of curves as you suggest. My feeling is that such a curve family will all lie within the precision range of a particular measurement.

(3) Mass 107 would always have a certain component due to molecular ion contribution. If this component is small and constant, it is sometimes better to accept this, rather than to sacrifice intensity; this is what happens when an offset voltage is used. Experience with your specimens has shown us that, even at low silver concentrations, a good deal of $^{107}Ag^{+}$ intensity is available. Consequently, we can use an offset voltage +(~ 75 volts) to suppress molecular ion contributions preferentially. In the most recent series we ran with an offset voltage in Series 2 and 3. I favour the use of an offset voltage particularly to protect the silver intensity from changes in the intensity of the neighbouring cadmium peaks.

4. Under our present FA conditions beam rastering would indeed reduce crater edge effects. However a raster of at least 125x125 um would be required to be effective, given our present experience. This would increase the uncertainty in the area analyzed. At this stage in the contract I would not recommend switching over to this mode, since our objective has been to analyze smaller areas.

Edge effects

5. The primary spot size is determined from inspection of Secondary ion image. Its diameter during the analysis was 40-50 um and was circular. New apertures are required about every three months. We change the apertures when we feel that the spot size and shape is detrimental to the analysis.

We will be discussing all this with Steve Chrisoulis.

Best regards
yours very truly

N. S. Mc Intyre

Energy, Mines and Resources Canada
Research and Technology
Canada Centre for Mineral and Energy Technology,
555 Booth Street,
Ottawa, Canada
K1A 0G1

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555, rue Booth,
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Your file Votre référence

Our file Notre référence

BY COURIER

C-14

February 20, 1985

Dr. N.S. McIntyre
Technical Director
Surface Science
Western Centre for Interdisciplinary
Studies in Chemical Physics
University of Western Ontario
London, Ontario
N6A 3K7

Dear Stewart:

I acknowledge the Feb. 8 letter of Darlene Johnston. I agree with her conclusion that the W-A standards should be used for the reanalysis of the Block B sphalerites. However, I have some reservations.

No. 603 is the same as No. 30 (see my letter of Dec. 11, 1984). When analysed in Block B it does not lie on the W-A calibration curve. Why not? Is it because it is possibly inhomogeneous or is it because there are effects we don't understand arising out of a different polished section? It worries me. I would also add that Ag for No. 35 on the W-A curve is also off the curve (Fig. 1).

I have made a few notes on Figs. 1 & 2. Also enclosed are the raw data sheets.

I assume that you noted my P.S. in my January 29 letter so that we finish this contract work before the 31st March.

I shall be out of town to March 4.

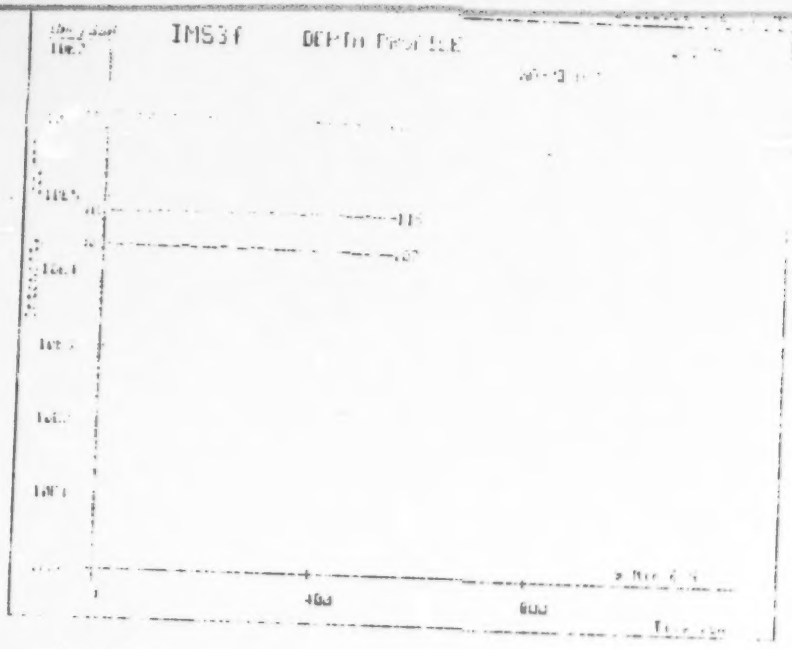
Best regards.

Louis J. Cabri

LJC/tj

Canada

C-14 (cont'd)



INJECTION: 35-M-1
DATE: 1 05
PRIMARY IONS : OXYGEN
SECONDARY IONS :
BEAM TIME: 500.00E-10 sec.

PRIMARY CURRENT : 20 nA/0 nA
HIGH VOLTAGE : 11.0 KILOVOLTS
RASTERING : 4 MICRONS
TRANSFER OPTICS : 150 MICRONS
FIELD APERTURE : 0 - 0
COLLECTOR APERTURE : 4 - 1

Normal

MASS NUMBER : 88.00 COUNT TIME : .00 OFFSET VOLTAGE : .0
M.R.P.: 51.72 1

TIME SEC	COUNT	TIME SEC	COUNT	TIME SEC	COUNT	TIME SEC	COUNT
	1.07753	21	1301390		[illegible]	42	[illegible]
2.	[illegible]	42	1319565	105	[illegible]	63	[illegible]
3.	1.43260	63	1333275	126	[illegible]	84	[illegible]
4.	1. [illegible]	84	1342[illegible]	147	[illegible]	105	[illegible]
5.	1. [illegible]	105	1348652	168	[illegible]	126	[illegible]
6.	1.09715	126	1356516	189	[illegible]	147	[illegible]
7.	1. [illegible]	147	1367081	210	[illegible]	168	[illegible]
8.	1.15056	168	1381755	231	[illegible]	189	[illegible]

MASS NUMBER : 107.00 COUNT TIME : .00 OFFSET VOLTAGE : .00
M.R.P.: 48.32

TIME SEC	COUNT	TIME SEC	COUNT	TIME SEC	COUNT	TIME SEC	COUNT
1.	.5[illegible]	29	20393	57	.11706	85	.[illegible]
2.	.[illegible]	57	20[illegible]	85	.13336	113	.[illegible]
3.	.[illegible]	85	20[illegible]	113	.14[illegible]	141	.[illegible]
4.	.[illegible]	113	20[illegible]	141	.15[illegible]	169	.[illegible]
5.	.[illegible]	141	20[illegible]	169	.16[illegible]	197	.[illegible]
6.	.[illegible]	169	20[illegible]	197	.17[illegible]	225	.[illegible]
7.	.[illegible]	197	20[illegible]	225	.18[illegible]	253	.[illegible]
8.	.[illegible]	225	20[illegible]	253	.19[illegible]	281	.[illegible]

MASS NUMBER : 11.00 COUNT TIME : .00 OFFSET VOLTAGE : .00
M.R.P.: 4.43

TIME SEC	COUNT	TIME SEC	COUNT	TIME SEC	COUNT	TIME SEC	COUNT
1.	[illegible]	14	67085	51	66423	88	66[illegible]
2.	67179	104	68393	102	67946	139	69[illegible]
15.	[illegible]	104	69009	192	69176	230	70[illegible]
2.9	[illegible]	145	69541	243	71013	280	15[illegible]
3.0	70[illegible]	195	71277	333	70533	41	70766
36.	70542	246	71259	404	72104	421	71110
4.9	[illegible]	156	70550	474	70497	516	71270
50.	[illegible]	527	69736	545	69423	0	0

.....TOTAL COUNTS IN PROFILE.....

88	39735277
107	717495
11	2150460

PROFILE TIME 546.000 SEC.
NUMBER CYCLE: 31.000

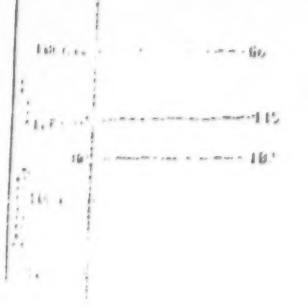
TOTAL COUNTS CORRECTED FOR DUT. CYCLE

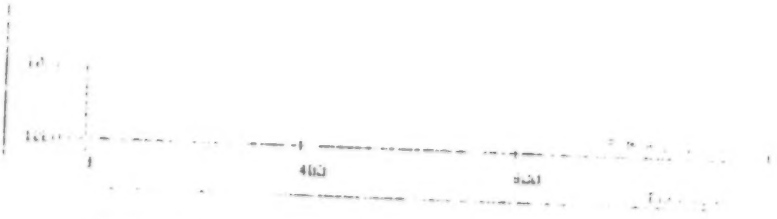
MASS	TOTAL COUNT
88	640575990
107	2313360
11	[illegible]

C-14 (cont)

TIME in S. CYCLE : 1.0
H.V.VOLTAGE: 0.0
TIME PERIOD (Sec): 150m

111534 DEPTH PROFILE

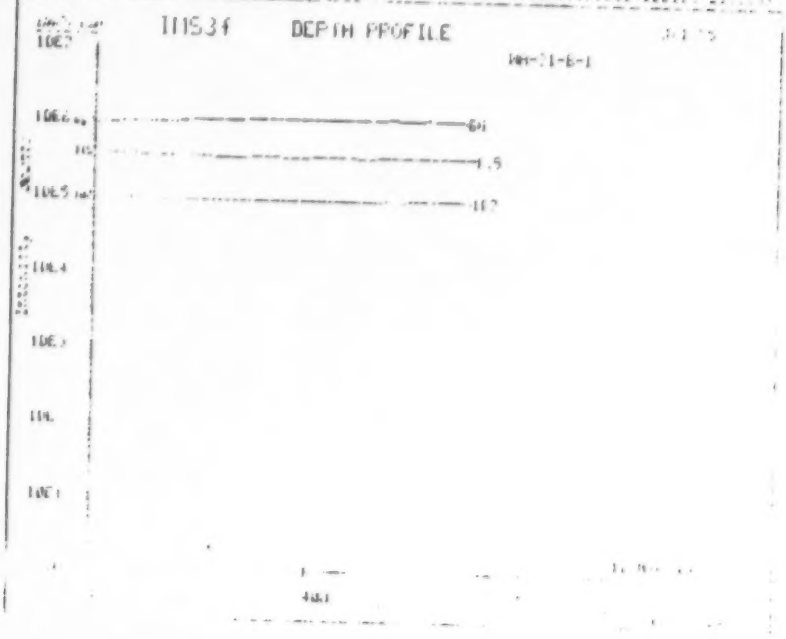




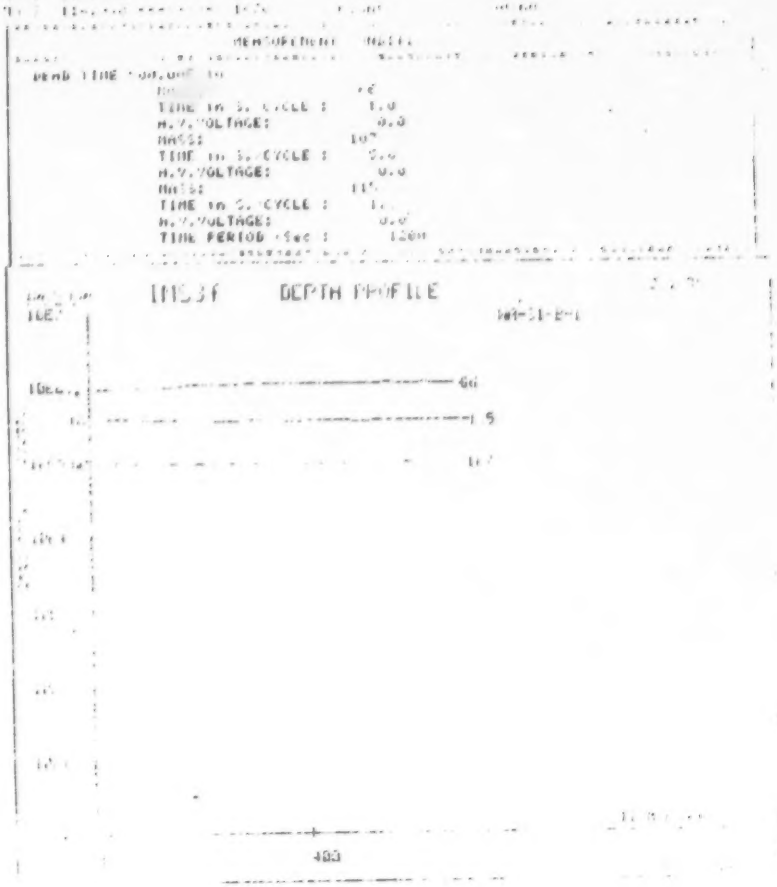
NANO AMP
KILOVOLT.
MICRONS
150 MICRONS

C-14 (cont'd)

DMEN PROGRAM
DEVELOPMENT
DEAD TIME 500.00E-10
TIME in % CYCLE :
TIME in % CYCLE :
TIME in % CYCLE :
TIME PERIOD (sec) :
120m


111534
DEPTH PROFILE
MM-11-6-1
6u
1.5
107

C-14 (cont'd)


MEMBRANE
TIME in % CYCLE :
M.V.VOLTAGE:
MASS:
TIME in % CYCLE :
M.V.VOLTAGE:
MASS:
TIME in % CYCLE :
M.V.VOLTAGE:
TIME PERIOD (Sec :
DEPTH PROFILE
60
15
483

Set 1

3/1/85

Raw data - 20 nA

FA 1 CA 3

No offset

stds wk -30 → 35

0.0

11

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

C-15

May 17, 1985

Dr. Louis J. Cabri
Canada Centre for Mineral and Energy Technology
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis:

I am enclosing the results of new SIMS measurements of the silver and indium concentrations in sphalerites contained in block WB. All measurements were made under the following conditions:

Field Aperture	1
Contrast Aperture	3
Primary O^- current	20nA (non-rastered)
Primary voltage	-11.8
Offset voltage	75 volts

Two independent series of measurements were made about a month apart, each carried out by a different analyst. In each case, the series of standards from block ~~WB~~ WA was measured at the beginning of the operating day. The first series, run on 29/03/85, was finished in one day and is called Run A. The second series stretched out over two days (26/04/85 and 29/04/85), necessitating two seperate calibrations; data obtained on these two days is labelled as Run B or Run C.

The ion currents collected for ^{107}Ag, ^{115}In, ^{66}Zn and ^{54}Fe are plotted as a function of time on the raw data sheets provided (please return as usual). Ratios of $^{107}Ag/^{66}Zn$, $^{107}Ag/^{54}Fe$, and $^{115}In/^{66}Zn$ are also printed out. The portion of the data used for further calculations varied with the stability of the silver and indium signals. In general, however, the final 10-50% of values were chosen and these were averaged. These values are given in Tables 1(a) (Run A) and 1(b) (Runs B and C) for both samples and standards. The ratio of $^{54}Fe/^{66}Zn$ is also shown in Table 1(a) for Run A.

The values from the standards are plotted in Figure 1 for Ag/Zn and In/Zn ratios and in Figure 2 for Ag/Fe ratios. Previous data is included as Figures 3 and 4. In Figure 1 the values obtained in each run can be

compared to the calibration plots reported in the letters of 11/01/85 and 08/02/85, and the agreement is seen to be reasonable. In Figure 2, the standard values of Ag/Fe show a particularly good linear correlation with known concentration. However the curve obtained has a definitely different intercept than that reported in our previous letters. Thus, for calculating the Ag and In values using a Zn reference, the previously-obtained calibration curves were used. However, for calculating Ag values using an Fe reference, the calibration curve used is based on values obtained in this present work.

Most of the unknown specimens analyzed in block WB were those previously analyzed by us (under old SIMS conditions) and also analyzed by PIXE. These results are compared in Table 2 for silver values and in Table 3 for Indium values. My immediate conclusions are:

(I) Silver

(1) Agreement between the two recently-completed SIMS studies is only fair. In only six out of fourteen cases, do the final results fall within a facter of two of each other. Some of this variance may be specimen-related. For example, specimen WB-3A gave a series of results widely varying in concentration, and gave evidence of changing silver ion currents during the measurement.

(2) Agreement between SIMS silver values from Runs B or C and PIXE is somewhat better. Nine of the fourteen specimens gave results from the two techniques which lie within a factor of two of each other. By contrast, only four of the SIMS results obtained under "old" conditions meet this critereon.

(3) The use of iron instead of zinc as a reference signal results in a poorer correlation between SIMS-derived silver concentrations and those obtained by PIXE (see Table 2). If the data for Runs B/C be compared for measurements made using zinc and iron references the decidely poorer agreement of the iron-based data can be seen. In the unknown sphalerites, iron concentrations appear to vary by a facter of four (see column D in Table 1(a)) and if the silver is, in some way, less associated with the iron than the zinc component, then we might expect a poorer correlation. In the standards which you prepared, however, it appears that the iron/silver correlation is very good better than zince/silver.

(4) In general, the poorest correlations between SIMS and PIXE data came from specimens WB-3 and WB-4. From the depth profile data obtained during these runs, as well as that sent earlier, we believe that these specimens are particularly heterogeneous.

(II) Indium

(1) The indium SIMS data also shows the best correlation between Runs B/C and the PIXE measurements. Eleven out of thirteen measurements fall within the facter of two critereon used above.

C-15 (cont.

(2) The indium SIMS data obtained under the "old" conditions definitely gives higher values than do the SIMS runs made under the revised operating conditions. We believe that molecular ion contributions were interfering severely with the In^+ signal under the old conditions where voltage offset was not used.

Let me know your thoughts on all this before I summarize our findings in the form of a final report.

Yours very truly

N. S. Mc Intyre

Table 1(a)

Ion Intensity Ratios - Run A

Date of Run	Specimen	Spectrum Number	Silver Standard Concentration (ppm)	A $\frac{I_{107}}{I_{66}}$	B $\frac{I_{107}}{I_{54}}$	C $\frac{I_{115}}{I_{66}}$	D $\frac{I_{54}}{I_{60}}$	Ag Conc. (ppm) (from A)	In Conc. (ppm) (from C)	Comments
27/03/85	WA-30-A	WA-30-A-10	1100	6.0 E-3	3.6 E-2	7.5 E-2	1.7 E-1	-	-	
	WA-30-A	WA-30-A-11	1100	7.5 E-3	3.7 E-2	8.4 E-2	2.1 E-1	-	-	
	WA-32-A	WA-32-A-11	270	2.7 E-3	1.1 E-2	2.1 E-2	2.4 E-1	-	-	
	WA-32-A	WA-32-A-10	270	2.7 E-3	1.1 E-2	2.3 E-2	2.4 E-1	-	-	
	WA-32-B	WA-32-B-10	270	2.5 E-3	1.1 E-2	2.2 E-2	2.2 E-1	-	-	
	WA-35-A	WA-35-A-11	16	1.9 E-4	7.4 E-4	1.3 E-3	2.7 E-1	-	-	
	WA-35-A	WA-35-A-10	16	1.9 E-4	6.5 E-4	1.2 E-3	2.8 E-1	-	-	(1)
	WB-24-A	WB-24-A-10	-	1.9 E-4	3.2 E-4	5 E-5	6 E-1	30	<1	
	WB-22-A	WB-22-A-10	-	3.6 E-4	4.5 E-4	1.3 E-1	8.1 E-1	55	1700	
	WB-20-A	WB-20-A-10	-	1.1 E-3	4.3 E-3	6.6 E-2	2.4 E-1	175	920	
	WB-19-A	WB-19-A-10	-	4.2 E-3	2.0 E-3	2.5 E-1	2.1 E-1	640	3200	
	WB-18-A	WB-18-A-10	-	2.3 E-3	3.7 E-4	3.3 E-1	6.1 E-1	360	4500	
	WB-14-A	WB-14-A-10	-	1.4 E-4	1.9 E-4	7.3 E-3	7.6 E-1	20	110	(2)
	WB-13-A	WB-13-A-10	-	5.0 E-4	6.1 E-4	7.5 E-2	8.3 E-1	80	1100	
	WB-10-A	WB-10-A-10	-	1.7 E-4	1.7 E-4	2.1 E-2	1.1 E-0	28	310	
	WB- 7-A	WB- 7-A-10	-	1.7 E-4	3.9 E-4	8.2 E-4	4.3 E-1	28	15	
	WB- 5-A	WB- 5-A-10	-	2.4 E-4	3.7 E-4	1.1 E-4		35	3	
	WB- 4-A	WB- 4-A-10	-	9.6 E-4	1.7 E-3	7.5 E-3	5.3 E-1	155	120	
	WB- 4A-A	WB- 4A-A-10	-	2.0 E-3	3.7 E-3	8.2 E-3	5.5 E-1	330	130	(2)
	WB- 3-A	WB- 3-A-10	-	2.7 E-3	5.8 E-3	1.2 E-2		280	260	(2)
	WB- 1-A	WB- 1-A-10	-	1.2 E-4	1.9 E-4	2.6 E-3	6.6 E-1	17	43	

C-15 (cont'd)

(1) 2 additional calibration runs made with results identical to above.

(2) The silver intensities for these points were found to vary noticably immediately prior to or during the measurement.

Table 1(b)

Ion Intensity Ratios - Run B

Date of run	Specimen	Spectrum Number	Ag Standard Concentration (ppm)	A $\frac{I_{107}}{I_{66}}$	B $\frac{I_{107}}{I_{54}}$	C $\frac{I_{115}}{I_{66}}$	D $\frac{I_{54}}{I_{66}}$	Ag Conc. (ppm)	In Conc. (ppm)	Comments
26/04/85	WA-30	WA-30-A-20	1100	7.1 E-3	4.1 E-2	6.5 E-2	3.7 E-1	-	-	
	WA-31	WA-31-A-20	550	4.3 E-3	2.1 E-2	3.9 E-2	2.0 E-1	-	-	
	WA-32	WA-32-A-20	270	1.3 E-3	9.1 E-3	1.4 E-2	-	-	-	
	WA-33	WA-33-A-20	110	9.0 E-4	4.4 E-3	9.1 E-3	-	-	-	
	WA-33	WA-33-A-20	110	1.2 E-3	4.6 E-3	1.1 E-2	-	-	-	
	WA-35		16	1.9 E-4	7.5 E-4	1.4 E-3	-	-	-	
	WB- 1-A	WB- 1-A-20	-	1.2 E-4	1.9 E-4	2.2 E-3	-	17	17	
	WB- 3-A	WB- 3-A-20	-	3.4 E-3	8.4 E-4	9.5 E-3	-	580	150	(2)
	WB- 3-A	WB- 3-A-20	-	1.8 E-1	1.1 E-1	1.1 E-2	-	$>10^4$	170	
	WB- 3-B	WB- 3-B-20	-	2.2 E-4	2.3 E-4	1.0 E-2	-	32	160	
	WB- 3-C	WB- 3-C-20	-	4.7 E-3	4.1 E-2	1.1 E-2	-	810	170	(2)
	WB- 4-A	WB- 4-A-20	-	5.0 E-3	2.4 E-3	8.6 E-3	-	850	138	(2)
	WB- 4A-A	WBO-4A-A-20	-	1.5 E-3	2.7 E-3	6.9 E-3	-	240	115	
	WB- 5-A	WB- 5-A-20	-	1.9 E-4	2.2 E-4	1 E-4	-	30	2	
	WB- 6-A	WB- 6-A-20	-	3.5 E-2	1.4 E-1	1.5 E-3	-	6600	27	(2)
	WB- 7-A	WB- 7-A-20	-	8 E-5	2 E-4	8 E-4	-	<10	15	
	WB-18-A	WB-18-A-20	-	1.1 E-3	3.9 E-4	2.2 E-1	-	180	2800	

C-15 (cont'd)

Table 1(b) (Cond.)

Date of Run	Specimen	Spectrum Number	Ag Standard Concentration (ppm)	Ion Intensity Ratios - Run C A $\frac{I_{107}}{I_{66}}$	B $\frac{I_{107}}{I_{54}}$	C $\frac{I_{115}}{I_{66}}$	D $\frac{I_{54}}{I_{66}}$	Ag Conc. (ppm) (From A)	In Conc. (ppm) (From C)	Comments
29/04/85	WA-30-A	WA-30-A-30	1100	6.4 E-3	3.9 E-2	5.9 E-2	-	-	-	
	WA-32-A	WA-32-A-30	270	2.0 E-3	1.1 E-2	1.5 E-2	-	-	-	
	WA-33-A	WA-33-A-30	110	7.6 E-4	4.4 E-3	6.2 E-3	-	-	-	
	WB- 2-A	WB- 2-A-30	-	1.2 E-4	2.4 E-4	7.4 E-4	-	16	14	
	WB- 6-A	WB- 6-A-30	-	1.0 E-3	3.9 E-2	1.4 E-3	-	160	25	(2)
	WB- 7-A	WB- 7-A-30	-	6 E-5	1.3 E-4	7.2 E-4	-	<10	14	(2)
	WB- 8-A	WB- 8-A-30	-	8 E-5	1.9 E-4	1.3 E-2	-	12	200	
	WB-10-A	WB-10-A-30	-	5 E-5	7.5 E-5	1.3 E-2	-	<10	200	
	WB-11-A	WB-11-A-30	-	1.0 E-4	1.5 E-4	1.1 E-3	-	14	20	
	WB-13-A	WB-13-A-30	-	1.8 E-4	3.2 E-4	5.5 E-2	-	26	750	
	WB-15-A	WB-15-A-30	-	1.4 E-3	1.9 E-3	4 E-5	-	220	<1	(2)
	WB-18-A	WB-18-A-30	-	1.4 E-3	3.8 E-4	2.7 E-1	-	220	3400	
	WB-19-A	WB-19-A-30	-	2.8 E-4	7.2 E-4	1 E-1	-	42	1400	
	WB-20-A	WB-20-A-30	-	5.5 E-4	3.1 E-3	4.4 E-2	-	84	630	
	WB-22-A	WB-22-A-30	-	7.1 E-4	-	9.7 E-2	-	115	1350	
	WB-24-A	WB-24-A-30	-	1.3 E-4	2.5 E-4	6 E-5	-	19	<1	
	WB-24A-A	WB-24A-A-30	-	1.2 E-4	1.5 E-4	1 E-4	-	16	2	
	WB-25-A	WB-25-A-30	-	3 E-5	-	8.3 E-3	-	<10	134	

C-15 (cont'd)

Table 2 C-15(cond)

Silver in Sphalerite Data
-Summary and Comparison
with Previous Data

Specimen No.	SIMS Ag Concentrations (ppm) -Derived from Zn Ratios			PIXE Data (ppm)	SIMS Ag Concentrations(ppm) -Derived from Fe Ratios	
	Old Conditions	New Conditions Run A	New Conditions Run B or C		New Conditions Run A	New Conditions Run B or C
WB- 1-A	71	17	17 —	<12	5	5
WB- 3-A (3)	297	280	580	49	150	18
WB- 3-A (3)			7104			
WB-3B			32			
WB-3C			810			
WB-4A (3)	59	155	850	287	40	58
WB-4A-A (3)	110	330	240 —	329	94	65
WB- 5-A	48	35	30 —	28	94	51
WB- 6-A			6600			
WB- 7-A (3)	43	28	<10 —	<12	100	37
WB-10-A	37	28	<10	25	40	<1
WB-13-A	42	80	26 —	45	13	7
WB-14-A (3)	14	20	-	<12	<5	-
WB-18-A	41	360	220	<12	8	8
WB-19-A	43	640	42 —	72	46	16
WB-20-A	58	175	84	36	102	72
WB-22-A	51	55	115 —	111	10	-
WB-24-A	40	30	19	<12	<	<5

(3) - Ag values varied noticeably during measurements.

Table 3 C-15 (cond)

Indium in Sphalerite Data

-Summary and Comparison

with Previous Data

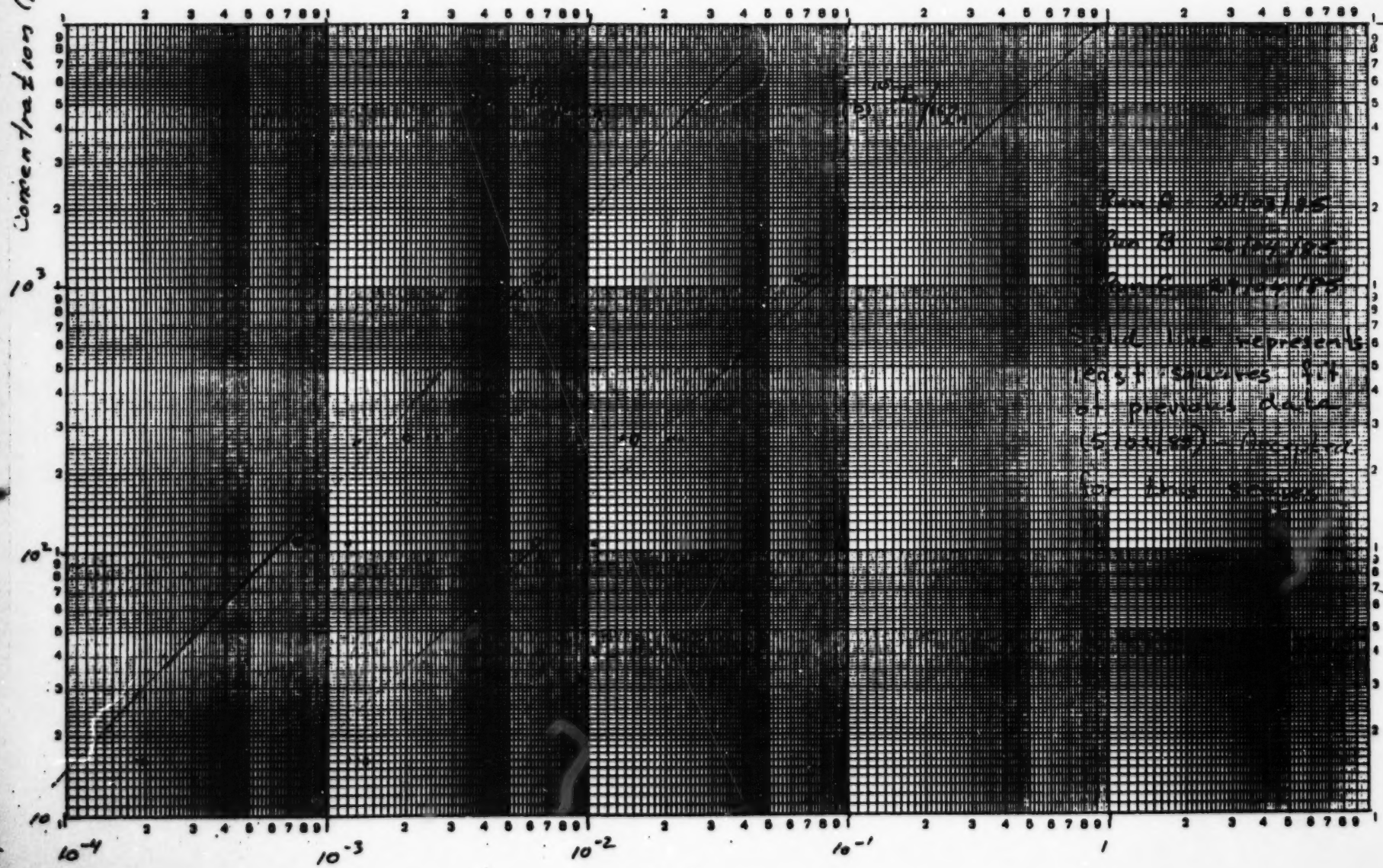
Specimen No.	SIMS In Concentrations (ppm) -Derived from Zn Ratios			PIXE Data (ppm)	E-Probe (ppm)
	Old Conditions	New Conditions Run A	New Conditions Run B or C		
WB- 1-A	93	43	17	<16	-
WB- 3-A	276	260	150	87	-
WB- 3-A			170		
WB-3B			160		
WB-3C			170		
WB- 4-A	284	120	138	31	-
WB-4A-A	674	130	115	35	-
WB- 5-A	18	3	2	<16	-
WB- 7-A	55	15	15	<16	-
WB-10-A	804	310	200	121	-
WB-13-A	2160	1100	750	618	775
WB-14-A	158	110	-	60	-
WB-18-A	8090	4500	2800	2060	2100
WB-19-A	5840	3200	1400	1640	1870
WB-20-A	1850	920	630	362	580
WB-22-A	4450	1700	1350	955	940
WB-24-A	17	<1	<1	<16	-

G-126 LOGARITHMIC 5 X 3 CYCLES
SPECIFY TRACING OR DRAWING PAPER

GRAPHIC CONTROLS CANADA LTD.
MADE IN CANADA

Figure 1 - $^{107}Ag/^{66}Zn$ and $^{115}In/^{66}Zn$ Calibrations

Conditions
FA 1 CA 3 20nA -.75KV offset



(-45 (cont)

G-125 LOGARITHMIC 5 X 3 CYCLES
SPECIFY TRACING OR DRAWING PAPER

GRAPHIC CONTROLS CANADA LTD.
MADE IN CANADA

Figure 2 $^{107}Ag/^{54}Fe$ Calibration

Conditions

FA 1 CA 3 20nA -75 eV offset

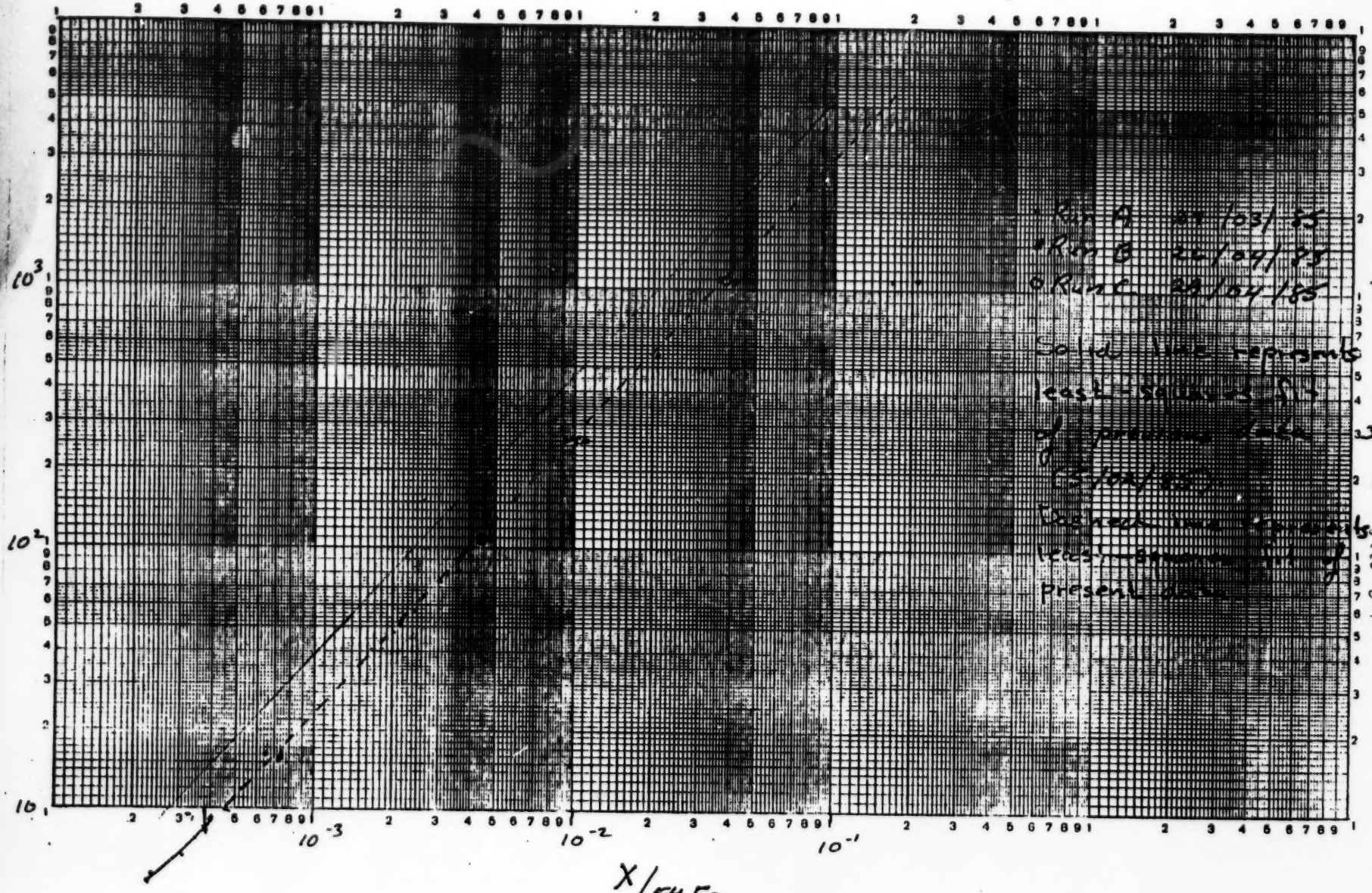


Fig 1

Q-125 LOGARITHMIC 5 X 3 CYCLES
SPECIFY TRACING OR DRAWING PAPER

GRAPHIC CONTROLS CANADA LTD.
MADE IN CANADA

5/2/85 FA1 CA3 20nA -75eV offset

× W-A stds
○ W-B 598
△ W-B 603
• W-B 622

• WA 27/3/85
□ 26/4/85

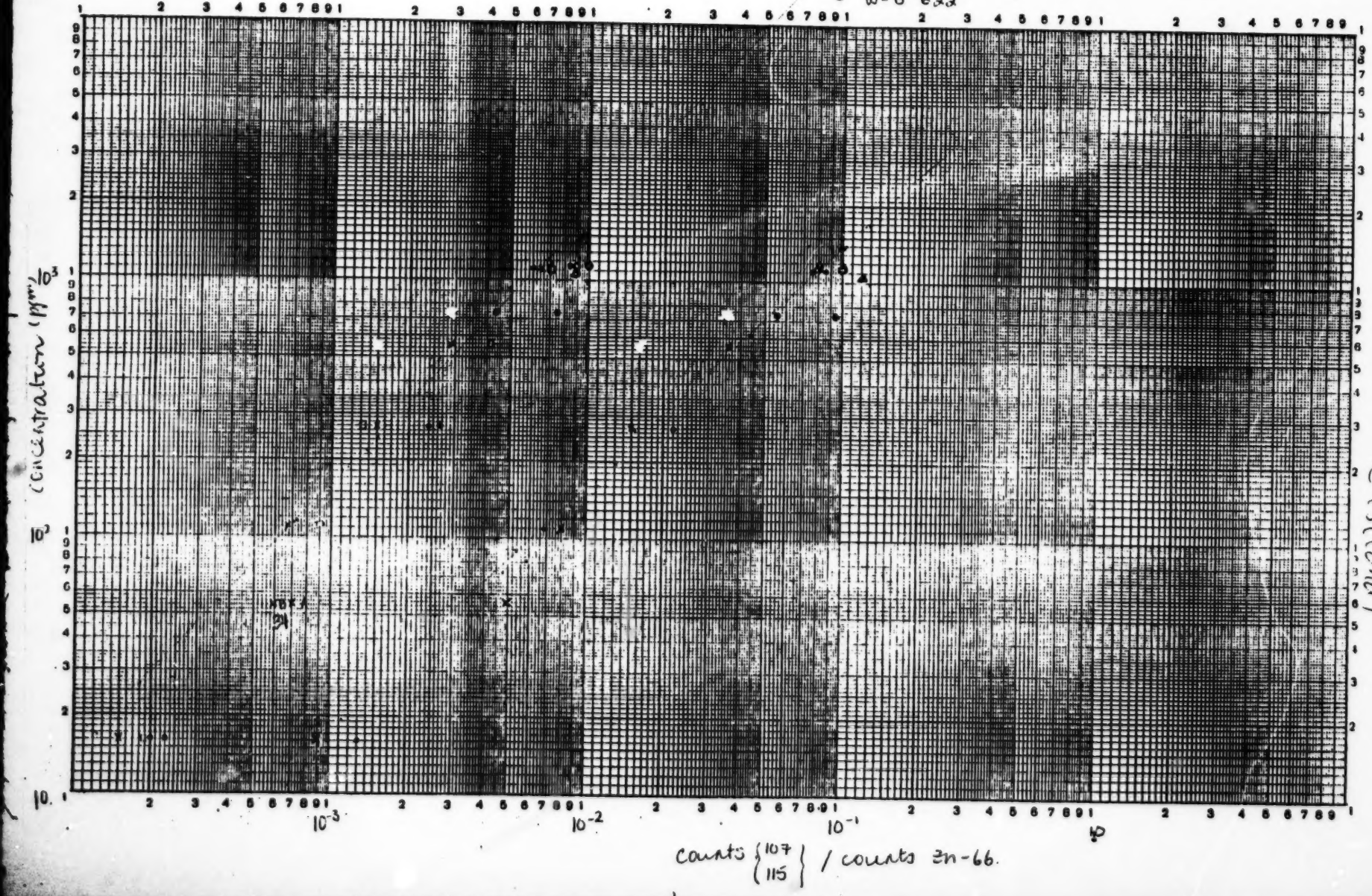


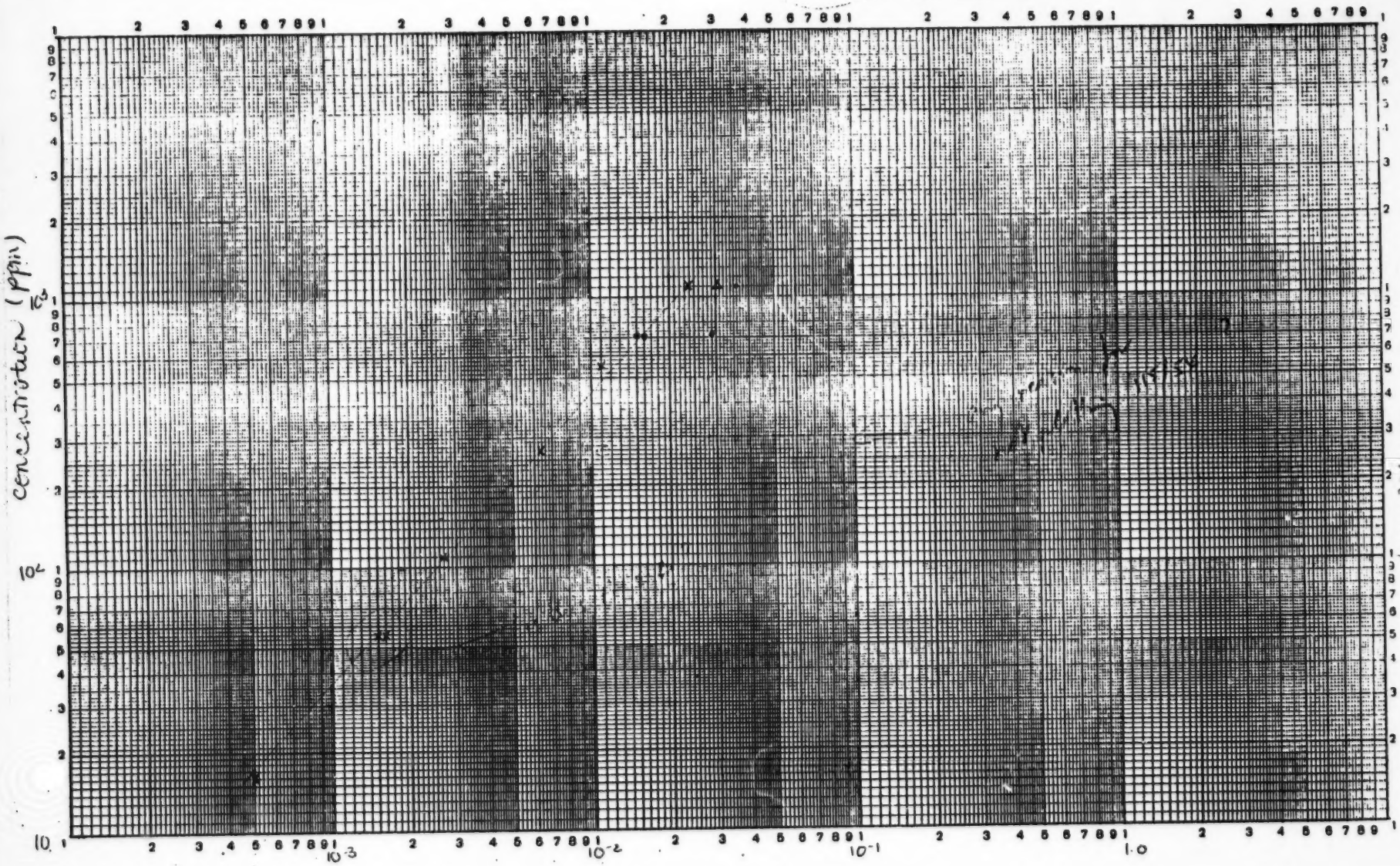
Fig A

G-125 LOGARITHMIC 6 X 3 CYCLES
SPECIFY TRACING OR DRAWING PAPER

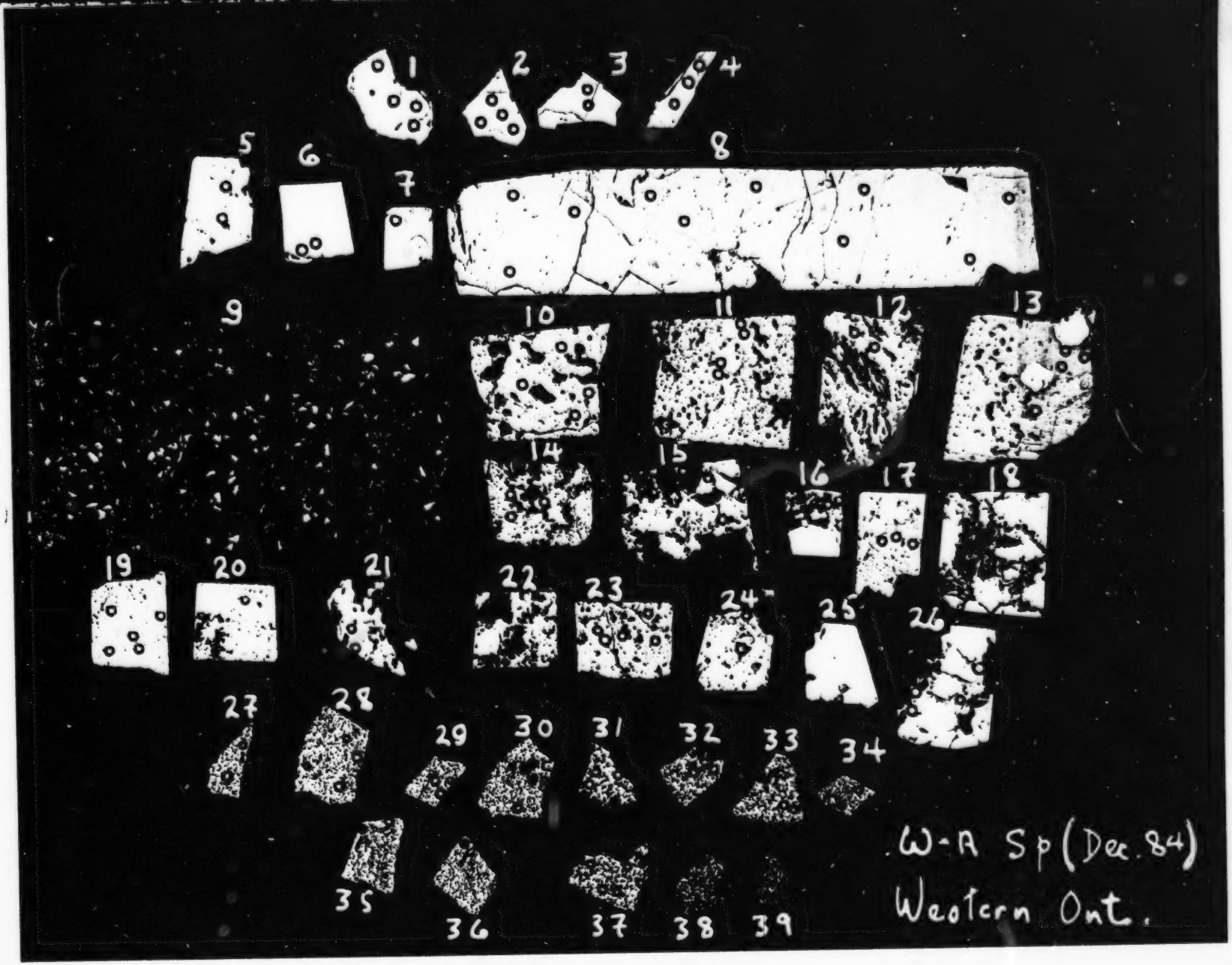
GRAPHIC CONTROLS CANADA LTD.
MADE IN CANADA

5/2/85 FA1 CH3 20nA -75eV offset

× WA stds.
o WB 598 – no Fe
Δ WB 603 = N+3
• WB 622



C-15(Cont'd)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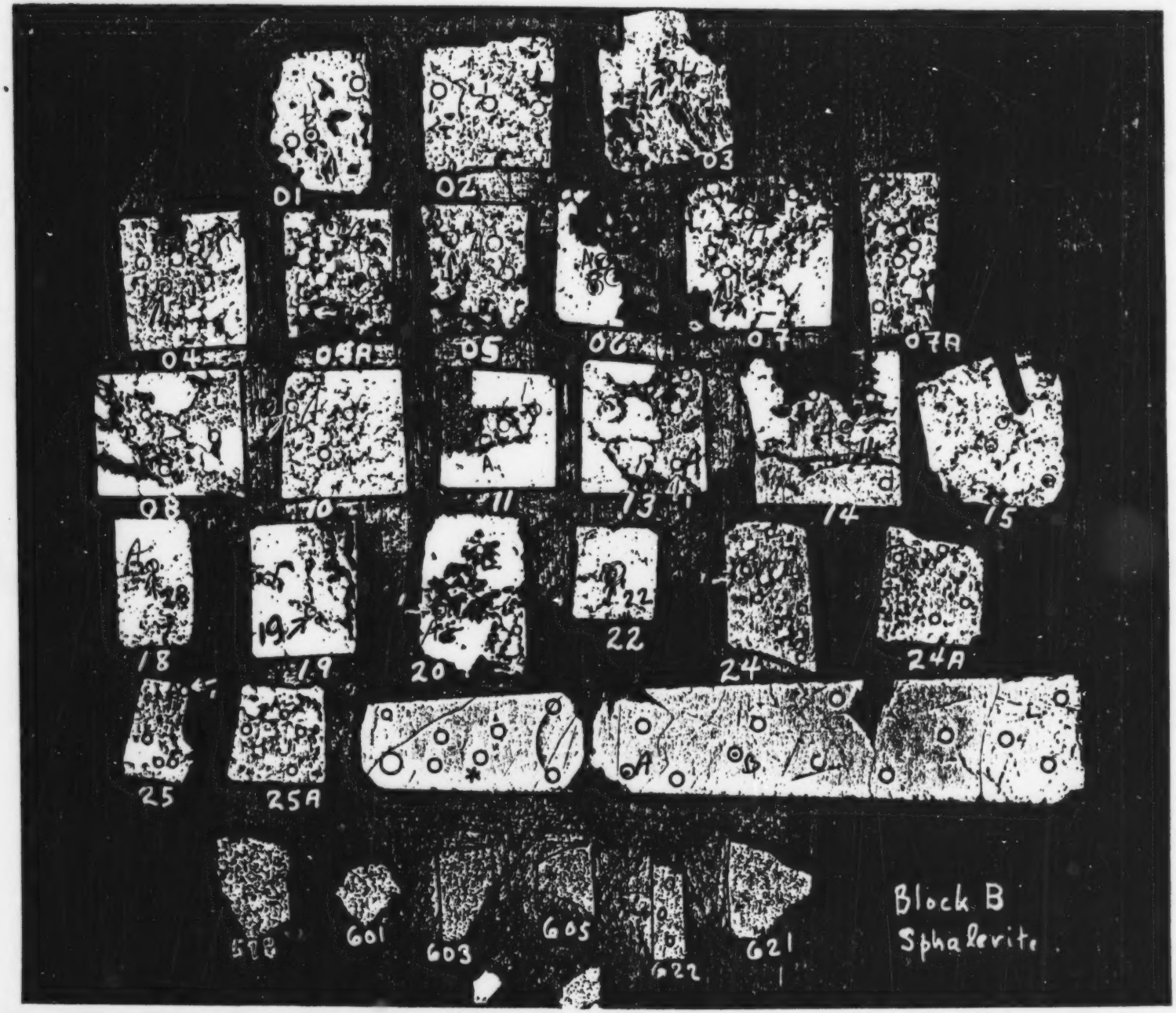
W-A Sp (Dec. 84)
Western Ont.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25A
Block B
Sphalerite

D

GALENA STUDIES

(September 1983 - February 1984)

Energy, Mines and Resources Canada
Research and Technology
Canada Centre for Mineral and Energy Technology,
555 Booth Street
Ottawa, Ontario
K1A 0G1

Énergie, Mines et Ressources Canada
Recherche et technologie
Centre canadien de la technologie des minéraux et de l'énergie,
555, rue Booth,
Ottawa (Ontario)
K1A 0G1

Your file Votre référence

Our file Notre référence

BY COURIER

D-1

April 5, 1984

Dr. N.S. McIntyre,
Technical Director,
Surface Science,
Western Centre for Interdisciplinary
Studies in Chemical Physics,
University of Western Ontario,
London, Ontario
N6A 3K7

Dear Stewart:

As discussed on the phone, enclosed is the second section (Block W-C) for analyses under the DSS contracts. This section was prepared for both galena (PbS) and chalcopyrite ($CuFeS_2$) but there is no point in doing the chalcopyrite as we have no series of standards.

I am still digesting the report you sent on the first set of sphalerite analyses and awaiting the additional data we talked about.

Best wishes.

Yours sincerely,

Louis J. Cabri

Encl.
LJC/tj

Canada

Block W-C (Galena/Chalcopyrite) 4/11/83

Sample #	2	/	Remarks
W-C	1	10	Five areas circled, could possibly do 2 to 3 spots or anal. in each one.
"	2	12	Six " " " " "
"	3	20	Ten " " " " "
"	4	3	Three areas circled, one anal. possible @ center of each one.
"	5	4	Two areas circled, could do 2 to 3 anal. in each one.
"	6	6	Three " " " "
"	7	8	Four " " " "
"	8	4	Two " " " "
"	9	6	Three " " " "
"	10	2	One " " " "
"	11	4	Two " " " "
"	12	4	Two " " " "
"	13		Synthetic std -569 which is straight PbS
"	14		" " -575 which contains 0.10 wt% Ag.
"	15		" " -580 " 0.05 "
"	16		" " -581 " 0.025 "
"	17		" " -582 " 0.010 "
"	18		" " -583 " 0.005 "
"	19		" " -584 " 0.001 "
"	20	6	Three areas circled, could possibly do 2 to 3 anal. in each one.
"	21	8	Four " " " "
"	22	3	Three areas circled, one anal. possible @ center of each one.
"	23		
"	24	8	Four areas circled, could possibly do 2 to 3 anal. in each one.
"	25	6	Three " " " "
		124	
min		136	
allb = 5 days			each = 3 days

D-1 (cont'd)

Surface Science Western

The Surface Science Laboratory, The University of Western Ontario,
Natural Sciences Centre, London, Ontario N6A 5B7
(519) 679-2233

D-2

October 18, 1984

Dr. L. J. Cabri
Canada Centre for Mineral and Energy Technology
Energy Mines and Resources Canada
555 Booth Street
Ottawa, Ontario
K1A 0G1

Dear Louis:

We are enclosing results of the analyses of grains of galena in block W-C as requested in your letter of April 5th.

Experimental Procedure

Mounting block W-C was sputter coated with 400 Å of gold. A primary beam of negative oxygen used for the analysis was mass filtered and focussed onto the analysis area as a 50 micron spot at a current of 50 na. Care was taken to position the beam in the centre of the pre-scored analysis areas on each specimen. Secondary ion currents were unstable in the near-surface areas of all specimens, and an anomalously high silver signal is detected. The beam was therefore allowed to dwell on the spot until satisfactory stability was reached, usually 10 to 20 minutes.

The computer was then set to measure the intensities of silver (mass 107) and indium (mass 115) and ratio these counts to those for lead (mass 204). This program sets up this routine as a cycle in which the background (mass 1.5) and each of the designated masses are counted for equal time periods (10 sec in this case). The resultant silver and indium intensities are then ratioed, after background subtraction, to the lead intensity. Five sequential cycles are run to comprise a block of data. Normally, two blocks of data were taken for each analysis, unless it appeared that, by the end of these two blocks, the beam was still analyzing a portion of the sample, judged to be a part of the anomolously high silver region, near the outer surface. In real time, the accumulation of two blocks of data takes 20 minutes.

At the beginning of each day the series of standards were run until at least three of the silver ratios agreed within 20% of the last ratio obtained for the same standard.

Results

The values for the standard series are given in Table 1 for each analysis

D-2 (cond)

date. These are plotted in Figure 1. Since no evolution of the values with time was noted, all data was assumed to be of equivalent validity, and a linear least-squares fit was determined from mean values, given in Table 1. From this regression, the uncertainty of the standard curve was such as to give a one standard deviation precision of $\pm$ 10 ug/g at 10 ug/g, $\pm$ 30 ug/g at 100 ug/g and $\pm$ 75 ug/g at 1000 ug/g. The raw data are enclosed for your inspection. Kindly return these for our records when you are finished with them.

Since a fairly large beam was used to increase the rate of penetration, it was not possible to obtain more than one analysis with a given area, particularly since at least one of the spots had to be centred. Based on the regression data from Figure 2, the silver concentrations were calculated for each cycle of data for each analysis. These are given in Table II. From this data it is easier to follow changes in silver concentration with time as the specimen is penetrated, and therefore to form some judgement as to whether the variations observed are still influenced by the anomalous surface layer, or are reflecting local changes in concentration. In some cases, a second run was started in the same spot (e.g. 1A2) where it appeared clear that a monotonic decrease in concentration appeared through the block of cycles.

The results of the last five cycles were averaged and are given in Table III. I believe, however, that close inspection of Table II tells more about the individual specimens, since in many cases the homogeneity (see 2F-1) or heterogeneity (see 2E-1) can be assessed.

We are returning block W-C and its photograph for your analyses. I look forward to your comments.

With best regards
yours very truly

N. S. Mc Intyre

N. S. Mc Intyre

Table 1

Average Ion Intensity Ratios Obtained During Analysis

of Reference Material in Block WC (Galena)

Date	Specimen	#13 blank	#14 1000ppm	#15 500ppm	#16 250ppm	#17 100ppm	#18 50ppm	#19 10ppm
une 7	-A-2,3,4	4.24×10^{-4}	3.70×10^{-2}	2.79×10^{-3}	9.88×10^{-3}	5.44×10^{-3}	1.28×10^{-3}	5.63×10^{-4}
uly 11	-A-10,11,12		3.26×10^{-2}		9.37×10^{-3}	6.56×10^{-3}		1.94×10^{-3}
ug 23	-A-20		4.51×10^{-2}			6.14×10^{-3}		1.18×10^{-3}
ept 6	-A-31		4.20×10^{-2}		1.07×10^{-2}		2.25×10^{-3}	
ept 7	-A-41		3.67×10^{-2}		1.30×10^{-2}		1.54×10^{-3}	
ept 13	-A-50		3.89×10^{-2}		1.34×10^{-2}		2.2×10^{-3}	
ept 25	-A-60		4.43×10^{-2}		1.23×10^{-2}		1.35×10^{-3}	
Mean Value ± Standard Deviation Over Dates Analyzed		(4.24×10^{-4})	3.95×10^{-2} ±.49	(2.79×10^{-3})	1.14×10^{-2} ±.19	6.05×10^{-3} ±.69	1.72×10^{-3} ±.52	1.23×10^{-3} ±.84

Conditions
Contrast Aperture - 4
Primary Current - 50 nA o⁻

Table II D-E (cont)

Silver Concentrations for WC Specimens

1-A-1

CNTS 107/204 *1000	PPM AG
17.78	431.
9.363	214.
6.621	143.
5.789	122.
5.	101.
4.408	86.
3.926	74.
3.493	62.
3.627	66.
3.142	53.

1-A-2

CNTS 107/204 *1000	PPM AG
1.841	20.
1.745	17.
1.364	8.
1.258	5.
1.189	3.

1-B-1

CNTS 107/204 *1000	PPM AG
13.97	333.
11.51	269.
9.187	209.
7.185	158.
5.474	114.
4.568	90.
3.786	70.
3.354	59.
2.901	47.
2.469	36.

1-B-2

CNTS 107/204 *1000	PPM AG
2.651	41.
2.179	29.
2.036	25.
1.978	23.
2.312	32.

D-2 (cont)

1-C-1

CNTS 107/204 *1000	PPM AG
4.449	87.
3.628	66.
2.688	42.
2.076	26.
1.541	12.
1.243	4.
1.017	<1.
.884	<1.
.9735	<1.

1-D-1

CNTS 107/204 *1000	PPM AG
6.242	133.
5.563	116.
5.019	102.
4.774	96.
4.08	78.
3.925	74.
3.746	69.
3.605	65.
3.497	63.

1-E-1

CNTS 107/204 *1000	PPM AG
2.986	49.
3.517	63.
3.412	60.
3.415	60.
2.991	50.
2.791	44.
2.464	36.
2.267	31.
2.925	48.
1.816	19.

2-A-1

CNTS 107/204 *1000	PPM AG
20.49	501.
15.83	381.
7.141	157.
5.575	116.
4.296	83.
4.29	83.
4.817	97.
5.282	109.
6.163	131.
5.451	113.

2-B-1

CNTS 107/204 *1000	PPM AG
4.476	88.
5.146	105.
4.808	96.
4.18	80.
4.161	80.
3.719	68.
8.446	190.
8.955	203.
8.956	203.
6.459	139.

2-B-2

CNTS 107/204 *1000	PPM AG
2.944	48.
2.983	49.
3.388	60.
3.002	50.
3.047	51.

2-C-1

CNTS 107/204 *1000	PPM AG
5.932	126.
7.468	165.
9.331	213.
8.791	199.
5.058	103.
4.81	96.
6.244	133.
7.479	165.
16.05	387.
24.05	593.

2-C-2

CNTS 107/204 *1000	PPM AG
13.88	331.
12.11	285.
7.058	154.
5.084	104.
3.969	75.

2-C-3

CNTS 107/204 *1000	PPM AG
3.51	63.
3.25	56.
3.446	61.
4.002	76.
3.717	68.

D-2 (cont'd)

2-D-1

CNTS 107/204 *1000	PPM AG
6.712	146.
6.14	131.
2.221	30.
5.655	118.
1.281	5.
4.863	98.
5.281	109.
6.559	142.
5.6	117.
4.36	85.

2-D-2

CNTS 107/204 *1000	PPM AG
5.431	113.
4.125	79.
4.072	77.
3.766	70.
4.123	79.
4.596	91.
3.837	71.
4.249	82.
4.515	89.
3.19	55.

2-E-1

CNTS 107/204 *1000	PPM AG
3.842	72.
3.597	65.
2.898	47.
2.843	46.
5.248	108.
7.939	178.
3.505	63.
2.212	30.
1.435	10.
1.226	4.

2-E-2

CNTS 107/204 *1000	PPM AG
1.948	23.
.6256	<1.
.5595	<1.
.5119	<1.
.4561	<1.

IJ-2 (cond)

2-F-1

CNTS 107/204 *1000	PPM AG
1.991	24.
1.807	19.
1.678	16.
1.724	17.
1.646	15.
1.555	12.
1.596	14.
1.567	13.
1.528	12.
1.524	12.

3-A-1

CNTS 107/204 *1000	PPM AG
2.366	33.
2.608	40.
2.095	26.
2.282	31.
1.922	22.
2.383	34.
1.942	22.
1.899	21.
2.354	33.
2.236	30.

3-B-1

CNTS 107/204 *1000	PPM AG
3.124	53.
2.554	38.
2.212	29.
1.881	21.
1.787	18.
1.559	13.
1.378	8.
1.456	10.
1.318	6.
1.232	4.

3-C-1

CNTS 107/204 *1000	PPM AG
5.772	121.
4.333	84.
3.776	70.
3.195	55.
2.948	48.
2.83	45.
2.749	43.
2.627	40.
2.507	37.
2.406	34.

D-2 (cont'd)

3-D-1

CNTS 107/204 *1000	PPM AG
6.958	152.
4.035	76.
2.709	42.
2.537	38.
1.931	22.
1.708	16.
1.588	13.
1.453	10.
1.408	9.
1.403	9.

3-E-1

CNTS 107/204 *1000	PPM AG
2.281	31.
1.986	24.
1.741	17.
1.555	12.
1.411	9.
1.263	5.
1.216	4.
1.183	3.
1.143	2.
1.034	<1.

3-F-1

CNTS 107/204 *1000	PPM AG
4.073	77.
3.295	57.
2.773	44.
2.505	37.
2.503	37.
2.039	25.
1.957	23.
1.812	19.
1.695	16.
1.597	14.

3-G-1

CNTS 107/204 *1000	PPM AG
2.637	40.
2.294	32.
2.117	27.
1.853	20.
1.939	22.
1.58	13.
1.578	13.
1.463	10.
1.352	7.
1.406	9.

D-2 (cont'd)

3-H-1

CNTS 107/204 *1000	PPM AG
3.84	71.
3.332	58.
3.124	53.
2.858	46.
2.71	42.
2.441	35.
2.357	33.
2.325	32.
2.273	31.
2.31	32.

3-I-1

CNTS 107/204 *1000	PPM AG
3.267	57.
3.143	53.
2.865	46.
2.684	42.
2.582	39.
2.521	37.
2.475	36.
2.4	34.
2.398	34.
2.396	34.

3-J-1

CNTS 107/204 *1000	PPM AG
3.799	70.
3.043	51.
2.792	44.
2.547	38.
2.349	33.
2.284	31.
2.133	27.
2.135	27.
2.023	25.
1.954	23.

4-A-1

CNTS 107/204 *1000	PPM AG
37.61	943.
37.61	943.
36.84	923.
37.28	934.
39.44	990.
42.81	1077.
42.54	1070.
41.55	1045.
42.38	1066.
43.08	1084.

D-2 (cont'd)

4-B-1

CNTS 107/204 *1000	PPM AG
20.16	493.
18.35	446.
18.63	453.
17.9	434.
16.99	411.
15.89	382.
15.78	380.
15.01	360.
13.98	333.
13.8	328.

4-B-2

CNTS 107/204 *1000	PPM AG
12.58	297.
11.29	264.
11.32	264.
11.2	261.
10.29	238.
9.941	229.
9.576	219.
9.235	211.
9.507	218.
8.065	180.

4-C-1

CNTS 107/204 *1000	PPM AG
30.07	748.
29.48	733.
29.64	737.
29.17	725.
29.08	723.
29.39	731.
28.91	718.
29.58	736.
29.9	744.
29.6	736.

5-A-1

CNTS 107/204 *1000	PPM AG
29.65	738.
29.53	734.
30.65	763.
27.7	687.
28.02	695.
30.09	749.
28.53	709.
28.33	703.
29.9	744.
28.7	713.

D-2 (cond)

5-B-1

CNTS 107/204 *1000	PPM AG
20.76	508.
20.69	506.
20.49	501.
20.39	499.
20.34	497.
20.21	494.
20.2	494.
20.15	492.
20.17	493.
20.25	495.

6-A-1

CNTS 107/204 *1000	PPM AG
64.34	1633.
67.89	1724.
59.1	1498.
54.15	1370.
54.89	1389.
55.25	1398.
63.23	1604.
54.16	1370.
55.58	1407.
50.16	1267.

6-A-2

CNTS 107/204 *1000	PPM AG
54.73	1385.
52.59	1330.
47.12	1188.
51.78	1309.
46.5	1172.
40.51	1018.
40.85	1027
36.29	909.
38.93	977.
41.95	1055.

6-B-1

CNTS 107/204 *1000	PPM AG
27.63	685.
27.12	672.
26.08	645.
27.74	688.
26.85	665.
26.28	651.
27.95	694.
26.71	662.
25.24	624.
25.32	626.

D-2 (cont'd)

6-C-1

CNTS 107/204 *1000	PPM AG
34.22	855.
32.84	820.
32.74	817.
33.75	843.
33.18	829.
34.85	872.
38.52	966.
33.65	841.
32.84	820.
33.02	824.

7-A-1

CNTS 107/204 *1000	PPM AG
37.87	950.
39.12	982.
42.21	1062.
41.51	1044.
40.59	1020.
40.41	1015.
43.11	1085.
41.58	1045.
43.67	1099.
41.87	1053.
42.34	1065.
38.71	971.
42.48	1069.
39.99	1004.
40.54	1019.
40.69	1022.
42.15	1060.
41.56	1045.
40.12	1008.
40.41	1015.

7-B-1

CNTS 107/204 *1000	PPM AG
63.89	1621
68.86	1749.
68.34	1736.
69.06	1755.
70.06	1780.
70.26	1786.
69.69	1771.
67.76	1721.
67.84	1723.
68.3	1735.

D-2 (cont'd)

7-C-1

CNTS 107/204	*1000	PPM AG
37.14		931.
38.08		955.
38.07		955.
37.61		943.
38.3		961.
39.03		980.
39.36		988.
38.73		972.
37.66		944.
37.99		953.

7-D-1

CNTS 107/204	*1000	PPM AG
45.44		1145.
45.84		1155.
45.17		1138.
45.2		1139.
44.22		1114
46.17		1164.
44.41		1118.
44.75		1127.
47.19		1190.
46.26		1166.

8-A-1

CNTS 107/204	*1000	PPM AG
55.		1392.
55.48		1404.
55.54		1406.
55.88		1414.
55.4		1402.
55.7		1410.
54.96		1391.
54.95		1390.
54.71		1384.
55.16		1396.

8-B-1

CNTS 107/204	*1000	PPM AG
61.26		1553.
61.06		1548.
61.96		1571.
61.86		1569.
62.07		1574.
62.59		1588.
61.84		1568.
62.43		1584.
63.05		1600.
62.94		1597.

D-21 (cont'd)

9-A-1

CNTS 107/204	*1000	PPM AG
28.69		713.
29.45		732.
29.82		742.
29.57		735.
30.49		759.
29.67		738.
31.09		775.
30.3		754.
30.31		755.
30.48		759.

D-2 (cont'd)

9-B-1

CNTS 107/204 *1000	PPM AG
29.58	736.7
28.65	712.
28.87	717.
27.75	688.
26.98	669.
26.41	654.
27.07	671.
26.82	664.
26.76	663.

9-C-1

CNTS 107/204 *1000	PPM AG
30.54	760.
30.15	750.
30.6	762.5
30.07	748.
30.33	755.
30.21	752.
31.01	773.
31.09	775.
30.48	759.
30.79	767.

10-A-1

CNTS 107/204 *1000	PPM AG
~~14.56~~	348.5
14.55	348.3
14.61	349.8
14.69	351.9
14.7	352.1
4.8	~~96.6~~
14.69	~~351.9~~

10-A-1

CNTS 107/204 *1000	PPM AG
14.56	348.
14.55	348.
14.61	349.
14.69	351.
14.7	352.
14.8	354.
14.69	351.
14.8	354.
14.8	354.

Date	Mean Value ± Standard Deviation Dates	Condition Code
June 7		
July 11		
Aug 23		
Sept 6		
Sept 7		
Sept 13		
Sept 25		

D-2 (cont'd)

11-A-1

CNTS 107/204 *1000	PPM AG
.7859	<1
.8443	<1
.6645	<1
.6707	<1
.5315	<1
.5361	<1
.5259	<1
.4699	<1
.4259	<1
.3658	<1

11-B-1

CNTS 107/204 *1000	PPM AG
2.73	43.
2.324	32.
2.299	32.
1.995	24.
1.929	22.
1.635	15
1.482	11
1.284	5.
1.212	4
1.177	3.

12-A-1

CNTS 107/204 *1000	PPM AG
.9001	<1
.5799	<1
.533	<1
.477	<1
.4687	<1
.4569	<1
.4103	<1
.3878	<1
.3649	<1
.3795	<1

12-B-1

CNTS 107/204 *1000	PPM AG
4.937	100.
4.195	81
3.363	59.
3.126	53.
2.705	42.
2.519	37.
2.261	31.
2.334	33
2.039	25.

D-21 (cont'd)

1.773	18.!
1.822	19.
1.501	11.
1.337	7.
1.221	4.
.9862	<1
.8698	<1
.8453	<1
.7905	<1
.7442	<1
.7551	<1

20-A-1

CNTS 107/204 *1000	PPM AG
.6839	<1
.41	<1
.4785	<1
.4144	<1
.3466	<1
.332	<1
.2847	<1
.2937	<1
.2686	<1
.2595	<1

20-B-1

CNTS 107/204 *1000	PPM AG
4.902	99.?
4.382	85.
3.132	53.
2.363	33.
1.917	22.2
1.405	9
1.363	7.
1.061	<1
1.066	<1
.9015	<1

20-B-2

CNTS 107/204 *1000	PPM AG
.284	<1
1.202	3.
.5865	<1
1.055	<1
.8819	<1
.8629	<1
.8068	<1
.6089	<1
.7763	<1
.5663	<1

D-2 (cont'd)

20-C-1

CNTS 107/204 *1000	PPM AG
4.065	77.
2.756	43.
2.141	28
1.518	11.
1.223	4.
.5535	<1
.9159	<1
.8172	<1
.7878	<1
.7111	<1

21-A-1

CNTS 107/204 *1000	PPM AG
.1363	<1
1.251	5.1
1274	32852.
1.241	4.8
1.254	5.1

21-A-1

CNTS 107/204 *1000	PPM AG
.1363	<1
1.251	5.
1.274	5.
1.241	4.
1.254	5.
1.214	4.
1.287	6
1.241	4.
1.204	3.
1.237	4.

21-B-1

CNTS 107/204 *1000	PPM AG
10.65	247.
7.893	176.
5.746	121.
4.485	88.
3.839	71.
3.203	55.
2.992	50
2653	41.
2.511	37.
2.339	33.

21-C-1

CNTS 107/204 *1000	PPM AG

D-21 (cont'd)

2.634	40.
2.315	32.
2.175	28.
2.034	25.
1.995	24.
1.867	20.
1.77	18.
1.715	17
1.615	14.
1.56	13

21-D-1

CNTS 107/204 *1000	PPM AG
5.141	105.
3.534	64
2.718	42.
2.313	32.
2.101	27
1.96	23.
1.792	19
1.709	16.
1.595	13.
1.563	13.

22-A-1

CNTS 107/204 *1000	PPM AG
18.6	452.
18.46	449.
18.35	446.
18.3	445.
18.21	442.
18.13	440.
3.577	65.
4.722	94.
18.08	439.
17.85	433.

22-B-1

CNTS 107/204 *1000	PPM AG
34.77	870.
35.55	890.
36.14	905.
36.31	909.
37.14	931.
37.77	947.
36.88	924.
37.04	928.
37.48	940
37.36	937

22-C-1

D-21 (cont'd)

CNTS 107/204 *1000	PPM AG
64.5400001	1638.4
55.37	1401.8
53.97	1365.6
60.5	1534.1
59.21	1500.9
56.27	1425
57.36	1453.1
56.56	1432.5
5.5	114.7
47.71	1204.1

22-C-1

CNTS 107/204 *1000	PPM AG
64.54	1638.
55.37	1401.
53.97	1365.
60.5	1534.
59.21	1500.
56.27	1425
57.36	1453.
56.56	1432.
52.5	1327.
47.71	1204.

23-A-1

CNTS 107/204 *1000	PPM AG
34.22	855.
34.12	853.
33.91	847.
34.14	853.
34.3	858
34.76	869.
33.91	847.
34.47	862.
34.29	857.
33.88	847.

23-B-1

CNTS 107/204 *1000	PPM AG
16.51	398.
16.44	397
16.41	396.
16.85	407.
16.23	391.
16.1	388.
16.12	388.
15.92	383.
15.75	379.

Da-2 (cont'd)

23-B-2

CNTS 107/204 *1000	PPM AG
12.92	306.
13.12	311.
12.92	306.
12.57	297.
12.16	286.
11.87	279.
12.63	298.
12.69	300.
13.39	318.
13.74	327.

23-C-1

CNTS 107/204 *1000	PPM AG
36.06	903.
34.55	864.
34.23	856.
34.06	851.
34.79	870.
33.25	830.
33.14	828
32.68	816.
33.5	837.
32.67	815

24-A-1

CNTS 107/204 *1000	PPM AG
56.57	1432.
58.61	1485.
59.35	1504.
62.01	1573.
62.71	1591.
64.77	1644.
66.5	1689
69.52	1766.
70.28	1786.
78.91	2009.

24-B-1

CNTS 107/204 *1000	PPM AG
83.76	2134.
81.01	2063.
77.92	1983.
78.35	1994.
28.59	710.
81.21	2068.
80.28	2044.
77.52	1973.
77.1	1962.
76.71	1952.

D-2 (cont'd)

24-C-1

CNTS 107/204 *1000	PPM AG
36.15	905.
36.35	910.
35.83	897.
36.47	914
36.02	902.
36.05	903.
35.89	899
36.35	910.
35.33	884.
36.54	915.

24-D-1

CNTS 107/204 *1000	PPM AG
51.99	1314.
53.89	1363.
52.05	1316.
52.8	1335.
54.09	1368.
51.74	1308.
53.58	1355.
52.01	1315
51.87	1311.
52.42	1325.

25-A-1

CNTS 107/204 *1000	PPM AG
46.74	1179
46.3	1167.7
46.39	1168.4
8.848	201.1
45.1	1136.7
6.077	129.6
4.39	86.1
4.439	87.3
4.456	87.8
4.334	84.6

25-A-1

CNTS 107/204 *1000	PPM AG
46.74	1179
46.3	1167.
46.33	1168.
8.84	200.
45.1	1136.
6.07	129.
43.9	1105.
44.39	1118.
44.56	1122.

43.34 1091.

D-21 (cont'd)

25-A-2

CNTS 107/204 *1000	PPM AG
46.79	1180.
48.41	1222.
48.45	1223.
46.53	1173.
46.33	1168.
45.54	1148.
45.91	1157.
44.96	1133.
45.23	1140.
45.04	1135.

25-B-1

CNTS 107/204 *1000	PPM AG
32.11	801.
32.09	800.
32.87	821.
32.3	806.
32.25	805.
32.07	800.
33.04	825.
32.34	807.
32.27	805.
32.06	800.

C-1

S 107/204 *1000	PPM AG
35.03	876.
33.63	840.
32.96	823.
32.05	799.
31.93	796.
32.39	808.
32.71	816.
33.25	830.
34.24	856.
33.25	830.

Table III - Block WC (Galena) Averaged Results

Date of Run	Specimen	counts 107/counts 204	ppm
23/08/84	1-A-2	1.48×10^{-3}	11
	1-B-2	2.23×10^{-3}	30
	1-C-1	1.03×10^{-3}	<1
	1-D-1	3.77×10^{-3}	70
	1-E-1	1.93×10^{-3}	23
	2-A-1	5.20×10^{-3}	107
	2-B-2	3.07×10^{-3}	52
	2-C-3	3.59×10^{-3}	65
	2-D-2	4.08×10^{-3}	78
	2-E-2	4.5×10^{-4}	<1
	2-F-1	1.55×10^{-3}	13
06/09/84	3-A-1	2.21×10^{-3}	30
	3-B-1	1.39×10^{-3}	9
	3-C-1	2.62×10^{-3}	40
	3-D-1	1.51×10^{-3}	12
	3-E-1	1.17×10^{-3}	3
	3-F-1	1.82×10^{-3}	20
	3-G-1	1.48×10^{-3}	11
	3-H-1	2.34×10^{-3}	33
	3-I-1	2.44×10^{-3}	36
	3-J-1	2.11×10^{-3}	27
	4-A-1	4.25×10^{-2}	1070
	4-B-2	9.26×10^{-3}	212
	4-C-1	2.95×10^{-2}	734
	5-A-1	2.91×10^{-2}	724
	5-B-1	2.02×10^{-2}	494
	6-A-2	3.97×10^{-2}	997
	6-B-1	2.63×10^{-2}	652
	6-C-1	3.46×10^{-2}	866
07/09/84	7-A-1	4.10×10^{-2}	1031
	7-B-1	6.88×10^{-2}	1748
	7-C-1	3.85×10^{-2}	966
	7-D-1	4.57×10^{-2}	1152
07/09/84	8-A-1	5.51×10^{-2}	1394
	8-B-1	6.26×10^{-2}	1588
	9-A-1	3.04×10^{-2}	757
	9-B-1	2.68×10^{-2}	664
	9-C-1	3.07×10^{-2}	765
	10-A-1	1.23×10^{-2}	350
	11-A-1	4.64×10^{-4}	<1
	11-B-1	1.36×10^{-3}	8
	12-A-1	3.99×10^{-4}	<1
13/09/84	12-B-1	8.01×10^{-4}	<1
	20-A-1	2.88×10^{-4}	<1
	20-B-1	7.24×10^{-4}	<1
	20-C-1	7.57×10^{-4}	<1
	21-A-1	1.24×10^{-3}	5
	21-B-1	2.74×10^{-3}	43
	21-C-1	1.71×10^{-3}	17
	21-D-1	1.72×10^{-3}	17
	22-A-1	1.84×10^{-2}	448
	22-B-1	3.73×10^{-2}	935
	22-C-1	5.62×10^{-2}	1423
25/09/84	23-A-1	3.42×10^{-2}	855
	23-B-2	1.28×10^{-2}	303
	23-C-1	3.31×10^{-2}	827
	24-A-1	7.00×10^{-2}	1779
	24-B-1	7.86×10^{-2}	2001
	24-C-1	3.60×10^{-2}	902
	24-D-1	5.23×10^{-2}	1322
	25-A-2	4.53×10^{-2}	1142
	25-B-1	3.24×10^{-2}	809
	25-C-1	3.32×10^{-2}	830

D-2 (cont)

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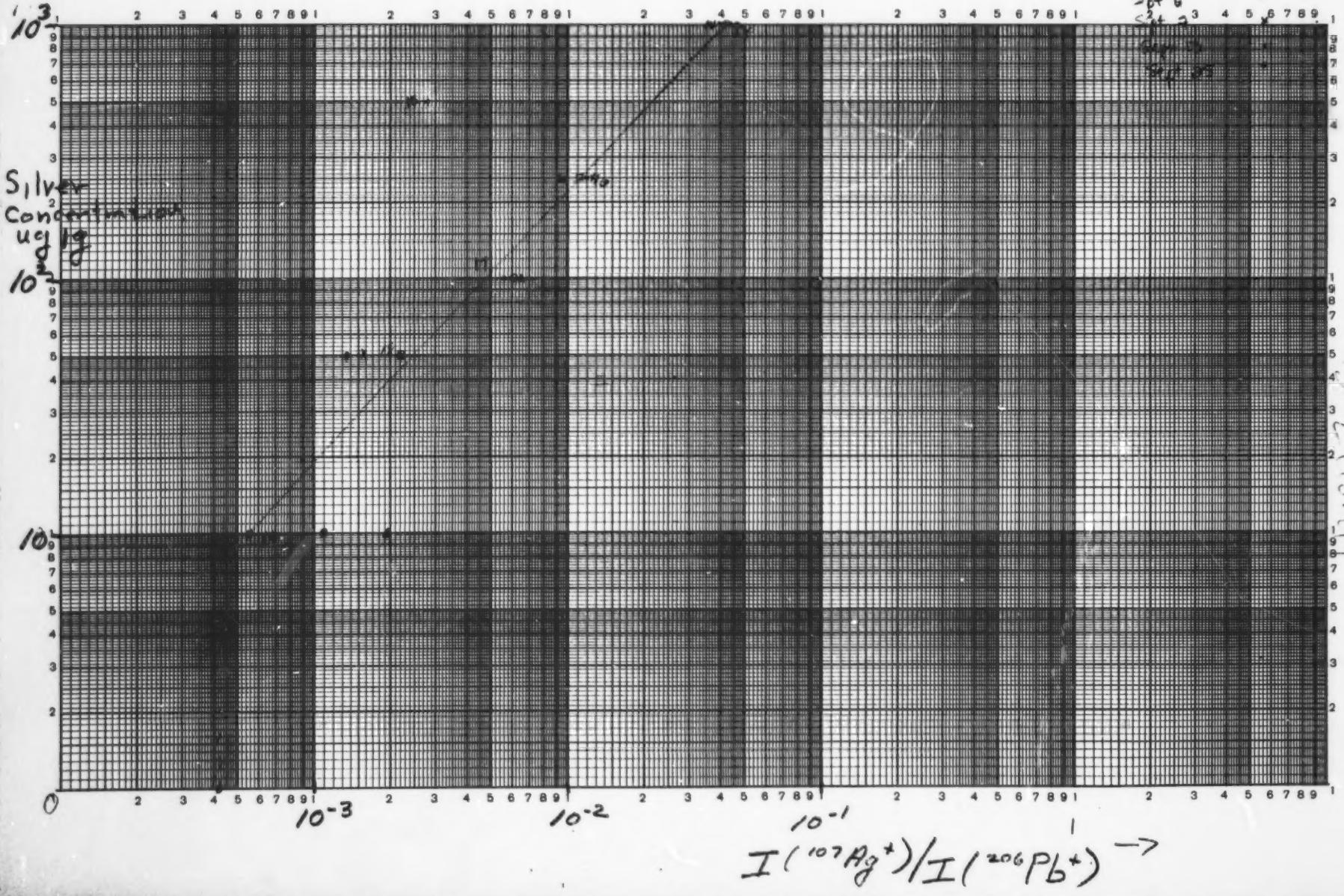
Figure 2 - Block WC (Galena) Calibration Points



D-2 (cont'd)

P-2 (cont'd)

DATA

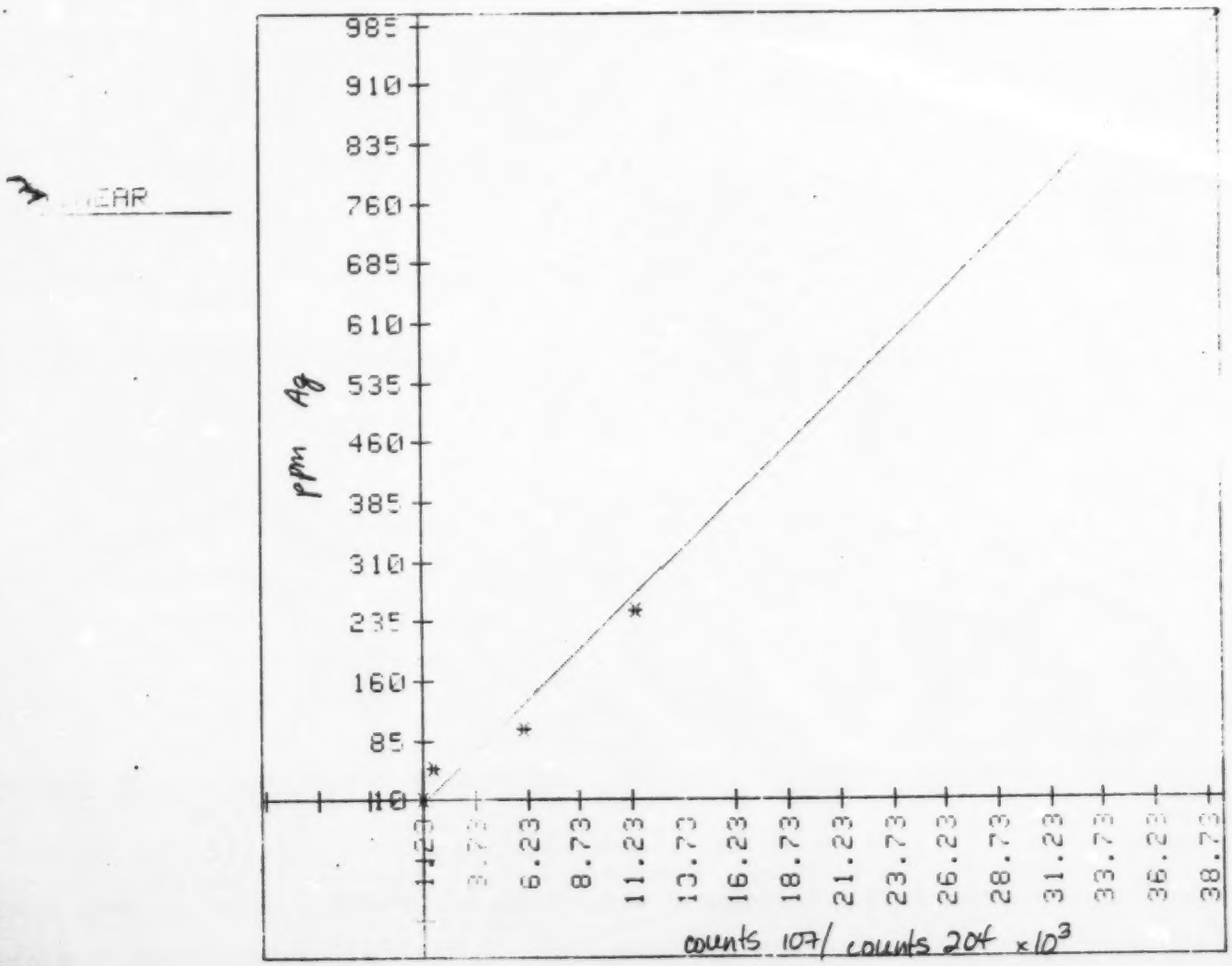
Point #1:	X=1.23	Y=10
Point #2:	X=1.72	Y=50
Point #3:	X=6.05	Y=100
Point #4:	X=11.4	Y=250
Point #5:	X=39.5	Y=1000

Figure 2

Regression Analysis of the mean ratios for the block WC Silver Standards

Linear model: Y=A+B*X
A=-27.184568569
B= 25.8083947053

Source	Df	SS	MS	F
Regression	1	675186.318	675186.318	883.104
Residual	3	2293.682	764.561	
Total	4	677480.000		

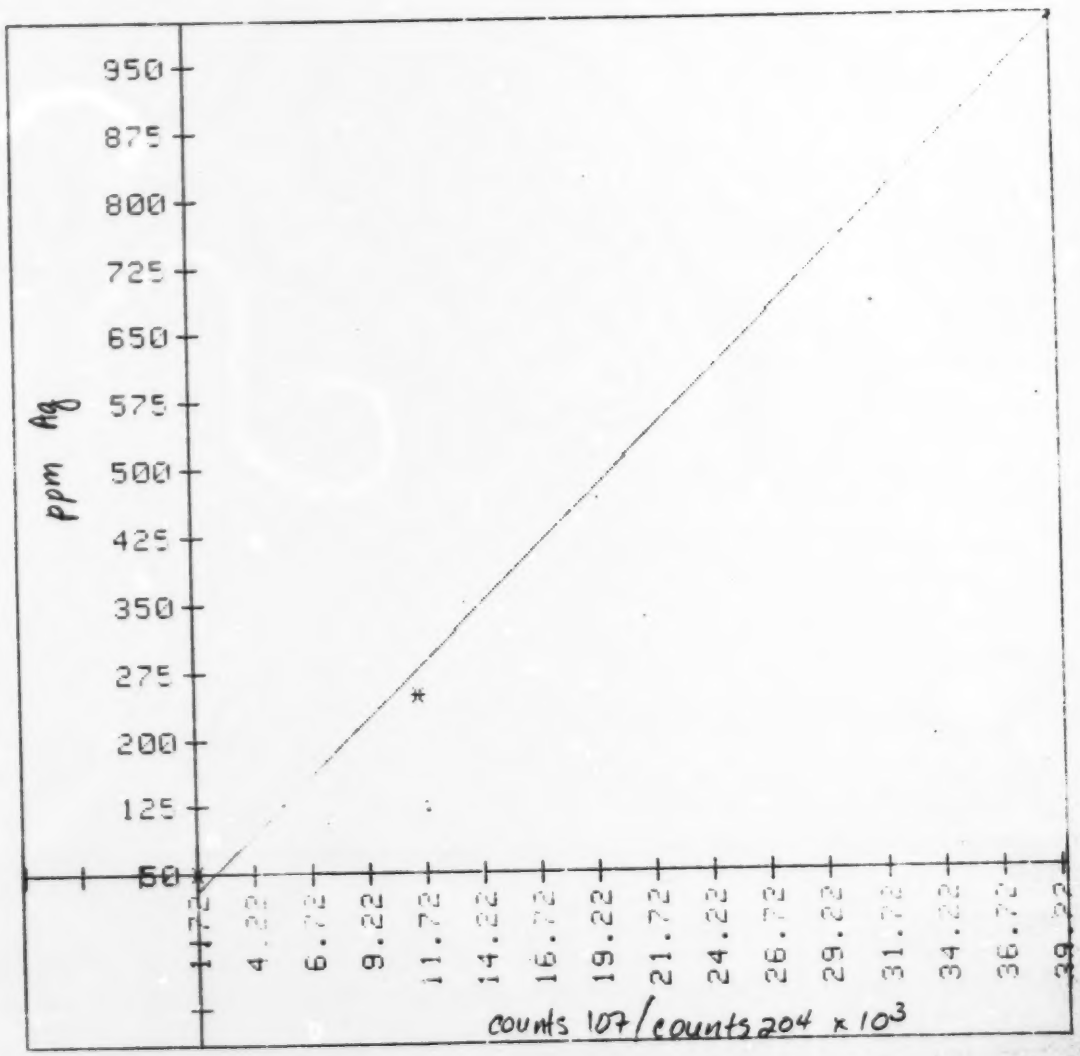


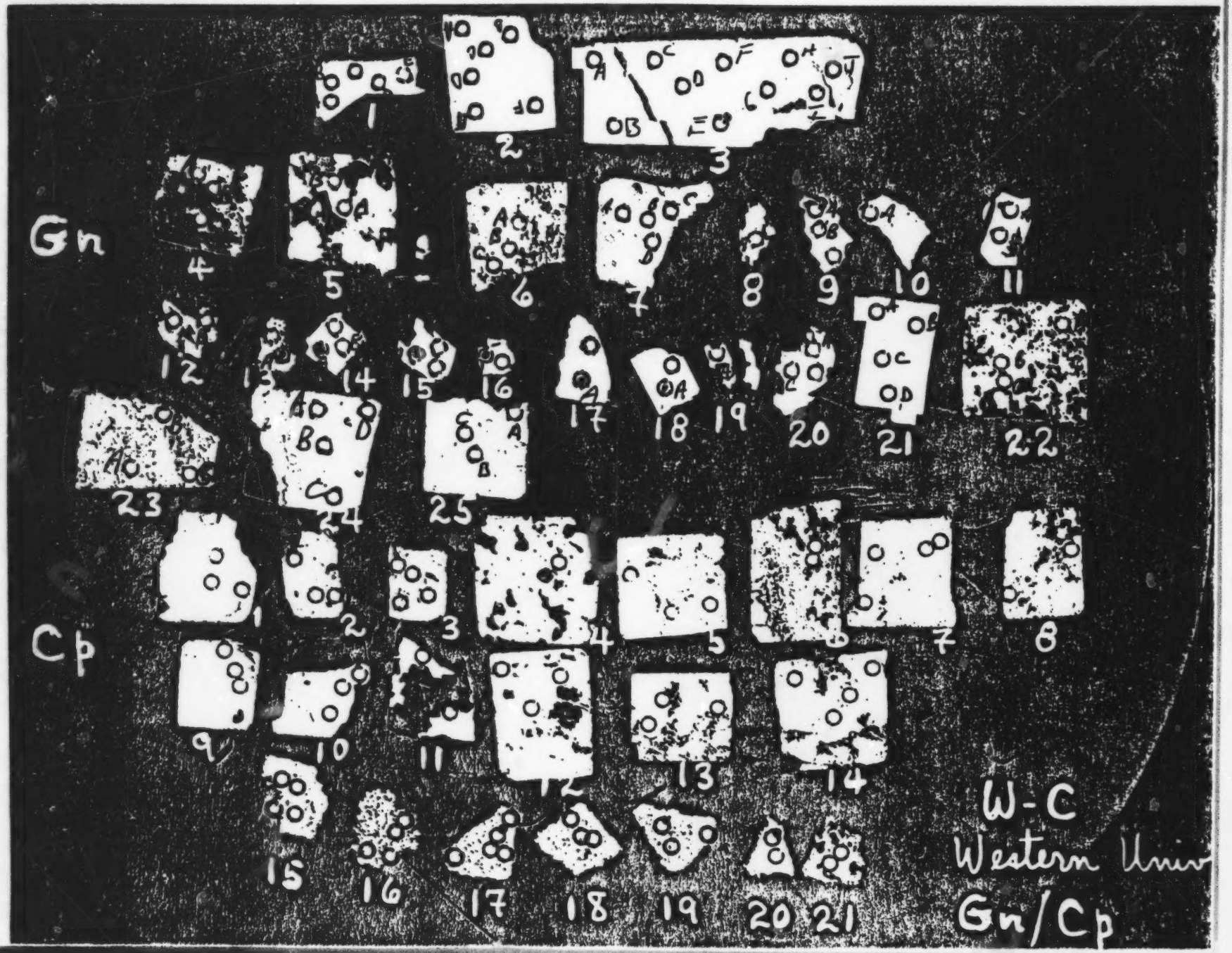
DATA

Point #1:	X=1.72	Y=50
Point #2:	X=11.4	Y=250
Point #3:	X=39.5	Y=1000

Linear model: Y=A+B*X
A=-13.789854313
B= 25.4916298544

Source	Df	SS	MS	F
Regression	1	500501.661	500502.661	429.983
Residual	1	1164.006	1164.006	
Total	2	501666.667		

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Cp
W-C
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